



FINAL REPORT

Cadmium Alternatives for High-Strength Steel

WP-200022

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14. ABSTRACT Testing was conducted for cadmium alternative coating systems IAW the "High Strength Steel Joint Test Protocol for Validation of Alternatives to Low Hydrogen Embrittlement Cadmium for High Strength Steel Landing Gear and Component Applications." Coatings suitable for full size components which were evaluated in this effort included sputtered Al, electroplated Al, LHE Zn-Ni, Acid Zn-Ni, and electroplated Sn-Zn which were compared against control coatings cadmium and IVD-Al. Repair coatings that were evaluated included brush plated Zn-Ni, brush plated Sn-Zn and spray/brush-applied SermeTel® 249/273. The control coating for repair was brush cadmium plating. The most promising candidates to replace cadmium plating included LHE Zn-Ni and electroplated Al which showed the most favorable balance of properties, although there are process specific reasons to consider the other coatings for certain applications. LHE Zn-Ni showed excellent corrosion resistance most similar to cadmium especially in the scribed (damaged) condition. In general, results showed that the aluminum based coatings perform well in corrosion fatigue, bend adhesion, paint adhesion, galvanic compatibility, and chemical strippability. The electroplated aluminum coating performed best in the various types of embrittlement and re-embrittlement tests conducted per the JTP.				
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Table of Contents

Note that original JTP section numbers are preceded by 5.1 in the JTP test descriptions section of this report (i.e. JTP Sec. 3.1.1 test descriptions are described in WP-0022 Final Report Sec. 5.1.3.1.1). Similarly, JPT Sec. 3.1.1 results and discussion are presented in WP-0022 Final Report Sec. 6.1.3.1.1.

Report Documentation Page.....	p. ii.
List of Acronyms and Abbreviations.....	p. vi.
List of Figures.....	p. viii.
List of Tables.....	p. x.
Acknowledgements.....	p. xii.
Executive Summary.....	p. xiii.
1.0 Introduction.....	p. 1.
1.1 Background.....	p. 1.
1.2 Objective of the Project.....	p. 6.
1.3 Regulatory Drivers.....	p. 9.
2.0 Technology Description.....	p. 12.
2.1 Cadmium and Alternative Coating Technologies.....	p. 12.
2.1.1 Ion Vapor Deposited Aluminum (IVD-Al) Process.....	p. 13.
2.1.2 Magnetron Sputtered Aluminum Process.....	p. 14.
2.1.3 Electroplated Aluminum Coating (AlumiPlate™) Process.....	p. 15.
2.1.4 Low Hydrogen Embrittlement (LHE) Zinc-Nickel (Zn-Ni) Processes.....	p. 16.
2.1.5 Tin-Zinc Electroplating Process.....	p. 17.
2.1.6 Brush Electroplated Cadmium Repair Coating Process and Alternatives.....	p. 17.
2.2 Technology Development.....	p. 18.
2.2.1 Ion Vapor Deposited Aluminum (IVD-Al) Technology Development.....	p. 18.
2.2.2 Magnetron Sputtered Aluminum Technology Development.....	p. 18.
2.2.3 Electroplated Aluminum (AlumiPlate™ Coating) Process Technology Development.....	p. 19.
2.2.4 Acidic Zinc-Nickel (Boeing) Electroplating Technology Development.....	p. 19.
2.2.5 LHE Zinc-Nickel (IZ-C17 Process) Technology Development.....	p. 19.
2.2.6 Tin-Zinc Electroplating Process Development.....	p. 20.
2.2.7 Brush Electroplated LHE Cadmium Development.....	p. 20.
2.2.8 Repair Coating Alternatives Development.....	p. 20.
2.3 Advantages and Limitations of these Alternative Technologies.....	p. 21.
3.0 Performance Objectives.....	p. 22.
4.0 Site/Platform Description(s)	p. 24.
4.1 Test Platforms/Facilities.....	p. 24.
4.1.1 LHE Zn-Ni (IZ-C17 process) Plating Demonstration.....	p. 24.
4.1.2 Magnetron Sputtered Aluminum Coating Demonstration.....	p. 25.
4.1.3 AlumiPlate™ Coating Demonstration.....	p. 25.
4.2 Present Operations.....	p. 25.
4.3 Site-Related Permits and Regulations.....	p. 26.
5.0 Test Design.....	p. 27.
5.1 Joint Test Protocol (JTP) Test Descriptions.....	p. 27.

5.1.3.1.1	Appearance (HSS JTP 3.1.1)	p. 32.
5.1.3.1.2	Throwing Power and Alloy Composition Uniformity (HSS JTP 3.1.2)	p. 32.
5.1.3.1.3	Strippability of Coatings (HSS JTP 3.1.3)	p. 34.
5.1.3.1.4	Electrochemical Galvanic Potential (HSS JTP 3.1.4)	p. 35.
5.1.3.2.1	Bend Adhesion (HSS JTP 3.2.1)	p. 37.
5.1.3.2.2	Wet Tape Paint Adhesion (HSS JTP 3.2.2)	p. 38.
5.1.3.3.1	Unscribed Salt Spray (Fog) Corrosion Resistance (HSS JTP 3.3.1).....	p. 39.
5.1.3.3.2	Scribed Salt Spray (Fog) Corrosion Resistance (HSS JTP 3.3.2)	p. 40.
5.1.3.3.3	Galvanic Corrosion Resistance (HSS JTP 3.3.3)	p. 40.
5.1.3.3.4	Fluid Corrosion Resistance (HSS JTP 3.3.4)	p. 43.
5.1.3.3.5	Corrosion Resistance of Scribed Painted Coatings (HSS JTP 3.3.5)	p. 44.
5.1.3.4.1	Run-on and Breakaway Torque (HSS JTP 3.4.1)	p. 46.
5.1.3.4.2	Torque Tension (HSS JTP 3.4.2)	p. 48.
5.1.3.4.3	Torque Tension for Corrosion Exposed Fasteners (HSS JTP 3.4.3)	p. 50.
5.1.3.5	Fatigue.....	p. 51.
5.1.3.5.1	Rotating Beam Fatigue (HSS JTP 3.5.1)	p. 51.
5.1.3.6	Hydrogen Embrittlement & Re-embrittlement.....	p. 52.
5.1.3.6.1	Hydrogen Embrittlement (HSS JTP 3.6.1)	p. 52.
5.1.3.6.2	Hydrogen Re-Embrittlement / In-Service Embrittlement (HSS JTP 3.6.2) ...	p. 55.
5.1.3.7.1	Repairability (HSS JTP 3.7.1)	p. 57.
5.1.4	U.S. Navy Added Requirements.....	p. 60.
5.1.4.1.1	Unscribed Cyclic SO ₂ Salt Spray (Fog) Corrosion Resistance.....	p. 60.
5.1.4.1.2	Scribed Cyclic SO ₂ Salt Spray (Fog) Corrosion Resistance.....	p. 61.
5.1.4.1.3	Cyclic SO ₂ Salt Spray (Fog) Corrosion Resistance of Scribed Painted.....	p. 62.
5.1.4.1.4	Corrosion Fatigue Test (HSS JTP 4.1.4)	p. 63.
5.1.4.3	Stress Corrosion Cracking and Susceptibility to H-E (HSS JTP Sec. 4.3)	p. 63.
5.1.5	U.S. Air Force Added Requirements.....	p. 65.
5.1.5.1	Acceptance criteria for Section 5.1.3.4.2 Torque Tension Test.....	p. 65.
5.1.6	U.S. Army Added Requirements.....	p. 65.
5.1.6.1	AMCOM In-Service Hydrogen Re-Embrittlement/SCC Test Plan.....	p. 65.
5.2	Field Testing Results.....	p. 66.
6.0	Performance Assessment.....	p. 67.
6.1	JTP Test Results and Discussion.....	p. 67.
6.1.3.1.1	Appearance (HSS JTP 3.1.1)	p. 71.
6.1.3.1.2	Throwing Power and Alloy Composition Uniformity (HSS JTP 3.1.2)	p. 72.
6.1.3.1.3	Strippability (HSS JTP 3.1.3)	p. 75.
6.1.3.1.4	Electrochemical Galvanic Potential (HSS JTP 3.1.4)	p. 77.
6.1.3.2	Adhesion.....	p. 78.
6.1.3.2.1	Bend Adhesion (HSS JTP 3.2.1)	p. 78.
6.1.3.2.2	Wet Tape Paint Adhesion (HSS JTP 3.2.2)	p. 83.
6.1.3.3	Corrosion Resistance (HSS JTP 3.3)	p. 85.
6.1.3.3.1	Unscribed Salt Spray (Fog) Corrosion Resistance (HSS JTP 3.3.1)	p. 85.
6.1.3.3.2	Scribed Salt Spray (Fog) Corrosion Resistance (HSS JTP 3.3.2)	p. 88.
6.1.3.3.3	Galvanic Corrosion Resistance (HSS JTP 3.3.3)	p. 89.
6.1.3.3.4	Fluid Corrosion Resistance (HSS JTP 3.3.4)	p. 94.
6.1.3.3.5	Corrosion Resistance of Scribed Painted Coatings (HSS JTP 3.3.5)	p. 100.

6.1.3.4	Lubricity (HSS JTP 3.4).....	p. 106.
6.1.3.4.1	Run-on and Breakaway Torque (HSS JTP 3.4.1).....	p. 106.
6.1.3.4.2	Torque Tension.....	p. 109.
6.1.3.4.3	Torque Tension for Corrosion Exposed Fasteners.....	p. 120.
6.1.3.5	Fatigue (HSS JTP 3.5).....	p. 120.
6.1.3.5.1	Rotating Beam Fatigue (HSS JTP 3.5.1).....	p. 120.
6.1.3.6	Hydrogen Embrittlement and Re-Embrittlement (HSS JTP 3.6)	p. 120.
6.1.3.6.1	Hydrogen Embrittlement (HSS JTP 3.6.1)	p. 120.
6.1.3.6.2	Hydrogen Re-Embrittlement (HSS JTP 3.6.2)	p. 125.
6.1.3.7.1	Repairability (HSS JTP 3.7.1)	p. 131.
6.1.4.0	Navy Added Corrosion Testing Results.....	p. 138.
6.1.4.1.1	Unscribed Cyclic SO ₂ Salt Spray (Fog) Corrosion Resistance.....	p. 140.
6.1.4.1.2	Scribed Cyclic SO ₂ Salt Spray (Fog) Corrosion Resistance.....	p. 143.
6.1.4.1.3	Cyclic SO ₂ Salt Spray Corrosion - Scribed Painted Coatings.....	p. 146.
6.1.4.1.4	Corrosion Fatigue Test (HSS JTP 4.1.4).....	p. 154.
6.1.4.3	Stress Corrosion Cracking and Susceptibility to H-E (HSS JTP Sec. 4.3)	p. 157.
6.1.5	U.S. Air Force Added Requirements.....	p. 158.
6.1.5.1	Acceptance Criteria for Section 5.1.3.4.2 Torque Tension Test.....	p. 158.
6.1.6	U.S. Army Added Requirements.....	p. 159.
6.1.6.1	AMCOM In-Service Hydrogen Re-Embrittlement/SCC.....	p. 159.
7.0	Cost Assessment.....	p. 162.
8.0	Implementation Issues.....	p. 163.
9.0	References.....	p. 165.
Appendix A	p. 167.
Appendix B	p. 168.
Appendix C	p. 177.
Appendix D	p. 178.

List of Acronyms and Abbreviations

AFRL	Air Force Research Laboratory
AFMC	Air Force Materiel Command
AIR	Aerospace Information Report
AL	Action Level
AMCOM	Army Aviation and Missile Command
AMS	Aerospace Material Specification
ANSI	American National Standards Institute
ARL	Army Research Laboratory
ASC/ENVV	Aeronautical Service Center / Environmental
ASTM	American Society for Testing and Materials
AT&L	Acquisition Technology and Logistics
BAC	Boeing Aircraft Company (Process Specification)
CBA	Cost Benefit Analysis
CPC	Corrosion Preventative Compound
CTC	Concurrent Technologies Corporation
DI	Deionized (Water)
ECA	Equivalent Circuit Analysis
EDM	Electric Discharge Machining
EIS	Electrochemical Impedance Spectroscopy
ESOH	Environmental Safety and Occupational Health
ESTCP	Environmental Security Technology Certification Program
EPA	(United States) Environmental Protection Agency
FRC	Fleet Readiness Center
HAFB	Hill Air Force Base
HazMat	Hazardous Material
H-E	Hydrogen Embrittlement
H-RE	Hydrogen Re-embrittlement
HSS	High Strength Steel
IAW	In Accordance With
ID	Inner Diameter
IDLH	Immediately Dangerous to Life and Health
ISE	In-Service Embrittlement
ISL	Incremental Step Load
ISSC	In-Service Support Center
IVD	Ion Vapor Deposition
JCAT	Joint Cadmium Alternatives Team
JG-PP	Joint Group on Pollution Prevention
JTP	Joint Test Protocol
JTR	Joint Test Report
K _{ISCC}	Threshold Stress Intensity for Stress Corrosion Cracking
K _{OSCC}	Threshold Stress Intensity for Stress Corrosion Cracking at Open Circuit
Kip	1000 pounds force
Ksi	kips per square inch

LDC	Liquid Development Company
LHE	Low Hydrogen Embrittlement
MERIT	Materials of Evolving Regulatory Interest Team
MIL-STD-	Military Standard
MIL-PRF-	Military Performance Specification
NACE	National Association of Corrosion Engineers (NACE International)
NAS	Naval Air Station
NAVAIR	Naval Air Systems Command
NAWCAD	Naval Air Warfare Center Aircraft Division
NDCEE	National Defense Center for Energy and Environment
NESDI	Naval Environmental Sustainability Development to Integration
NIEHS	National Institute of Environmental Health Sciences
NFS	Notch Fracture Strength
NIOSH	National Institute for Occupational Safety and Health
OCP	Open Circuit Potential
OEM	Original Equipment Manufacturer
OO-ALC	Ogden Air Logistics Center (U.S. Air Force)
OSHA	Occupational Safety and Health Administration
PAX	Naval Air Station Patuxent River, MD
PEL	Permissible Exposure Limit
PPE	Personal Protective Equipment
REACH	Regulation, Evaluation, Authorization and Restriction of Chemicals
ROHS	Reduction of Hazardous Substances
RSL	Rising Step Loading
SAE	Society of Aerospace Engineers
SCC	Stress Corrosion Cracking
SCE	Saturated Calomel Electrode
SEM	Scanning Electron Microscopy
SIFCO	Finishing Company
SLT	Step Loading Technique
TWA	Time Weighted Average
UTS	Ultimate Tensile Strength
WMTR	Westmoreland Mechanical Testing and Research
XRF	X-ray Fluorescence

List of Figures

Figure 1.	Coated Test Coupons Prior To Test – Primary Coatings	Pg. 8.
Figure 2.	Coated Test Coupons Prior To Test – Repair Coatings	Pg. 9.
Figure 3.	Typical Electroplating Operation.....	Pg. 12.
Figure 4.	Typical Vacuum Coating Deposition Chamber.....	Pg. 12.
Figure 5.	Vacuum Coating System at HAFB / OO-ALC.....	Pg. 13.
Figure 6.	Example of Industrial Sputter Coating Apparatus.....	Pg. 14.
Figure 7.	AlumiPlate™ Coating Process Line.....	Pg. 15.
Figure 8.	Zinc-Nickel Plating Process.....	Pg. 16.
Figure 9.	Representative Repair Coating Application Processes.....	Pg. 18.
Figure 10.	Initial Concept for LHE Zn-Ni Process line at Navy FRC-SE.....	Pg. 24.
Figure 11.	Process Schematic for Existing Cadmium Cyanide Electroplating.....	Pg. 26.
Figure 12.	Fixture for “Throwing Power” and Alloy Composition Test.....	Pg. 33.
Figure 13.	Assembly of Galvanic Corrosion Resistance Test Article.....	Pg. 41.
Figure 14.	Galvanic Corrosion Resistance Measurement.....	Pg. 42.
Figure 15.	Maximum Locking Torque and Breakaway Torque Test Setup.....	Pg. 46.
Figure 16.	Schematic Torque-Tension Test Fixture.....	Pg. 48.
Figure 17.	Hydrogen Embrittlement Test Equipment at NAS Patuxent River.....	Pg. 53.
Figure 18.	Representative Photo of Coated Panels Prior to Testing.....	Pg. 71.
Figure 19.	Photographs of Bend Adhesion Panels (Phase I).....	Pg. 80.
Figure 20.	Low Magnification SEM Images of Bend Samples.....	Pg. 81.
Figure 21.	Optical Images of Failure Modes of Bend Adhesion Specimen.....	Pg. 79.
Figure 22.	Representative Photos of Bend Adhesion Results for Repair Coatings.....	Pg. 83.
Figure 23.	Representative Photos of Wet Tape Adhesion Panels.....	Pg. 84.
Figure 24.	Photos of Unscribed Corrosion Resistance Panels After Exposure.....	Pg. 87.
Figure 25.	Photos of Scribed Corrosion Resistance Panels after Exposure.....	Pg. 89.
Figure 26.	Representative Photos of Salt Spray-Exposed Fixtures.....	Pg. 91.
Figure 27.	Representative Photos of Cyclic Corrosion-Exposed Fixtures.....	Pg. 94.
Figure 28.	Representative Photos of Coated Panels Prior to Test.....	Pg. 94.
Figure 29.	Cadmium Plated Panels after Fluid Immersion.....	Pg. 96.
Figure 30.	IVD-Aluminum Panels after Fluid Immersion.....	Pg. 97.
Figure 31.	LHE Zn-Ni Panels after Immersion.....	Pg. 97.
Figure 32.	Electroplated Al Panels after Immersion.....	Pg. 98.
Figure 33.	Sputtered Al Panels after Immersion.....	Pg. 98.
Figure 34.	Representative Painted and Scribed Corrosion Resistance Panels.....	Pg. 103.
Figure 35.	Graph of Maximum Locking Torque for the 3/8-inch Fasteners.....	Pg. 107.
Figure 36.	Graph of Breakaway Torque for 3/8-inch Fasteners.....	Pg. 108.
Figure 37.	Graph of Maximum Locking Torque for the 5/8-inch Fasteners.....	Pg. 108.
Figure 38.	Graph of Breakaway Torque for 5/8-inch Fasteners.....	Pg. 109.
Figure 39.	Torque-Tension Test Fixture.....	Pg. 110.
Figure 40.	Torque Tension Results for LHE Cd, 3/8-inch Fastener.....	Pg. 111.
Figure 41.	Torque Tension Results for Zn-Ni Coated 3/8-inch Fasteners.....	Pg. 112.
Figure 42.	Torque Tension Results for 3/8-inch Fasteners w/Electroplated Al.....	Pg. 113.
Figure 43(a).	Torque Tension Results for 3/8-inch Fasteners Coated w/Sputtered Al.....	Pg. 114.

Figure 43(b).	Torque Tension Results for 3/8-inch Fasteners Coated with IVD-Al.....	Pg. 115.
Figure 44.	Torque Tension Results for LHE Cd-coated 5/8-inch Fastener.....	Pg. 116.
Figure 45.	Torque Tension Results for 5/8-inch Fastener Coated with LHE ZnNi.....	Pg. 117.
Figure 46.	Torque Tension Results for 5/8-inch Fastener Coated with AlumiPlate™.....	Pg. 118.
Figure 47(a).	Torque Tension Results for 5/8-inch Fastener Coated with Sputtered Al.....	Pg. 119.
Figure 47(b).	Torque Tension Results for 5/8-inch Fastener Coated with IVD-Al.....	Pg. 120.
Figure 48.	Hydrogen Embrittlement Test Equipment.....	Pg. 121.
Figure 49.	Fracture Surface of Brush Zn-Ni Bar / 16.9 Hours at 75% NFS.....	Pg. 124.
Figure 50.	Hydrogen Re-Embrittlement Test Equipment	Pg. 125.
Figure 51.	Visual Depiction of Al Coating Density.....	Pg. 128.
Figure 52.	Representative Photos of Bend Adhesion Results for Repair Coatings.....	Pg. 132.
Figure 53.	Cross-sectional Images of Coating for Thickness Measurement.....	Pg. 133.
Figure 54.	Photos of Unscribed Corrosion Resistance Panels for Repair Coatings.....	Pg. 135.
Figure 55.	Photographs of Scribed Corrosion Resistance Panels for Repair Coatings...	Pg. 137.
Figure 56.	Unscribed Cyclic SO ₂ Salt Fog Corrosion Resist.....	Pg. 142.
Figure 57.	Unscribed Cyclic SO ₂ Salt Fog Corrosion Resistance (Ratings vs. Time)....	Pg. 143.
Figure 58.	Scribed Cyclic SO ₂ Salt Fog Corrosion Resistance.....	Pg. 145.
Figure 59.	Scribed Cyclic SO ₂ Salt Fog Corrosion Resistance (Ratings vs. Time).....	Pg. 146.
Figure 60.	Scribed, Painted Cyclic SO ₂ Salt Fog Corrosion Resistance (Cadmium).....	Pg. 148.
Figure 61.	Scribed, Painted Cyclic SO ₂ Salt Fog Corrosion (LHE Zn-Ni).....	Pg. 149.
Figure 62.	Scribed, Painted Cyclic SO ₂ Salt Fog Corrosion Resistance (IVD-Al).....	Pg. 150.
Figure 63.	Scribed, Painted Cyclic SO ₂ Salt Fog Corrosion (AlumiPlate™).....	Pg. 151.
Figure 64.	Scribed, Painted Cyclic SO ₂ Salt Fog Corrosion (Sputtered Al).....	Pg. 152.
Figure 65.	Scribed, Painted Cyclic SO ₂ Salt Fog Corrosion Ratings vs. Time.....	Pg. 153.
Figure 66.	Stress Life Curves.....	Pg. 155.
Figure 67.	Bare and Plated Fatigue in Air and 3.5% NaCl Solution.....	Pg. 156.
Figure 68.	Variation of K _{OSCC} and K _{ISCC} for Precracked vs. Un-Precracked 4340.....	Pg. 157.
Figure 69.	Variation of K _{OSCC} with V _{SCE} for Bare and Coated Specimens.....	Pg. 158.
Figure 70.	Army C-ring Specimens in the Salt Spray Cabinet.....	Pg. 159.

List of Tables

Table 1.	Timeline for Cadmium Alternatives Testing and Guidance Documentation.....	Pg. 5.
Table 2.	Coating Information for Cadmium Alternatives Tested.....	Pg. 7.
Table 3.	Advantages and Disadvantages of Each Alternative.....	Pg. 21.
Table 4.	Performance Objectives for the Alternative Coating Systems.....	Pg. 23.
Table 5.	Common Performance and Testing Requirements.....	Pg. 27.
Table 6.	Extended Performance and Testing Requirements.....	Pg. 29.
Table 7.	Test Facility Identification for Phases I/II JTP Test Execution.....	Pg. 30.
Table 8.	Test Specimen Codes and Substrate Descriptions.....	Pg. 31.
Table 9.	Test Methodology for Coating Appearance.....	Pg. 32.
Table 10.	Test Methodology for Throwing Power / Composition Testing.....	Pg. 34.
Table 11.	Test Methodology for Strippability Testing.....	Pg. 35.
Table 12.	Test Methodology for Electrochemical Galvanic Potential Testing.....	Pg. 37.
Table 13.	Test Methodology for Bend Adhesion Testing.....	Pg. 37.
Table 14.	Test Methodology for Wet Tape Paint Adhesion Testing.....	Pg. 39.
Table 15.	Test Methodology for Unscribed Salt Spray Testing.....	Pg. 39.
Table 16.	Test Methodology for Unscribed Salt Spray Testing.....	Pg. 40.
Table 17.	Recommended Parts List for Galvanic Assembly.....	Pg. 41.
Table 18.	Recommended Parts List for Galvanic Assembly.....	Pg. 43.
Table 19.	Test Methodology for Fluid Corrosion Resistance.....	Pg. 44.
Table 20.	Test Methodology for Scribed Painted Corrosion Resistance.....	Pg. 45.
Table 21.	Test Methodology for Run-on and Breakaway Torque.....	Pg. 47.
Table 22.	Test Methodology for Torque Tension.....	Pg. 49.
Table 23.	Test Methodology for Corrosion Exposed Torque Tension.....	Pg. 51.
Table 24.	Test Methodology for Hydrogen Embrittlement.....	Pg. 53.
Table 25.	Incremental Step Loading Protocols used in Phases I and II.....	Pg. 56.
Table 26.	Test Methodology for Hydrogen Re-Embrittlement.....	Pg. 57.
Table 27.	Initial Qualification Test Method for Brush Plating Repairs.....	Pg. 58.
Table 28.	Final Qualification Test Method for Brush Plating Repairs.....	Pg. 59.
Table 29.	Test Methodology for Unscribed Cyclic SO ₂ Corrosion Resistance.....	Pg. 61.
Table 30.	Test Methodology for Scribed Cyclic SO ₂ Corrosion Resistance.....	Pg. 61.
Table 31.	Test Methodology for Scribed Painted Cyclic SO ₂ Corrosion.....	Pg. 62.
Table 32.	Test Methodology for Fatigue Testing.....	Pg. 63.
Table 33.	Test Methodology for SCC Testing.....	Pg. 64.
Table 34.	JTP Performance Objective Ratings for Primary Coatings	Pg. 69.
Table 35.	Performance Objective Ratings for each Repair Candidate.....	Pg. 70.
Table 36.	Appearance of Primary Coatings.....	Pg. 71.
Table 37.	LHE Cadmium Panel Composition Results – Orientation #1.....	Pg. 72.
Table 38.	LHE Cadmium Panel Composition Results – Orientation #2.....	Pg. 72.
Table 39.	LHE Cadmium Panel Composition Results – Orientation #2.....	Pg. 72.
Table 40.	Electroplated Al Panel Composition Results – Horizontal.....	Pg. 73.
Table 41.	Electroplated Al Panel Composition Results – Open End Downward.....	Pg. 73.
Table 42.	Electroplated Al Panel Composition Results – Open End Upward.....	Pg. 74.
Table 43.	LHE Zn-Ni Panel Composition Results – Open End Upward.....	Pg. 75.

Table 44. LHE Zn-Ni Panel Composition Results – Open End Downward.....	Pg. 75.
Table 45. LHE Zn-Ni Panel Composition Results – Open End Horizontal.....	Pg. 75.
Table 46. Summary of Strippability Test Results.....	Pg. 76.
Table 47. H-E Test Results for Chemically Stripped and Recoated Specimens.....	Pg. 77.
Table 48. Open Circuit Potentials (OCP) Measured on SCC Test Specimens.....	Pg. 78.
Table 49. Bend Adhesion Test Results – Phase I.....	Pg. 79.
Table 50. Bend Adhesion Test Results.....	Pg. 80.
Table 51. Bend Adhesion Results for Repair Coatings.	Pg. 83.
Table 52. Averaged Wet Tape Adhesion Results.....	Pg. 84.
Table 53. Corrosion Resistance Results – Unscribed Panels.....	Pg. 86.
Table 54. Corrosion Resistance Results – Scribed Panels.....	Pg. 88.
Table 55. Change in Resistivity for ASTM B 117 Exposed Fixtures.....	Pg. 90.
Table 56. Change in Resistivity of Cyclic Corrosion Fixtures.....	Pg. 92.
Table 57. ASTM F 1110 Corrosion Severity Rating System.....	Pg. 95.
Table 58. Fluid Corrosion Resistance Test Results for Cd-plated Panels.....	Pg. 99.
Table 59. Corrosion Resistance Results – Painted and Scribed Panels.....	Pg. 102.
Table 60. ASTM B 117 Corrosion Resistance for Painted, Scribed Cd Alternatives.....	Pg. 105.
Table 61. GM9540P Corrosion Resistance for Painted, Scribed Cd Alternatives.....	Pg. 106.
Table 62. Test Method for Torque Tension.....	Pg. 110.
Table 63. Hydrogen Embrittlement Test Results, Primary and Repair Coatings (Phase I).....	Pg. 122.
Table 64. Hydrogen Embrittlement Reproducibility Test Results Coatings (Phase II).....	Pg. 124.
Table 65. Hydrogen Re-Embrittlement Test Results (Phase I).....	Pg. 126.
Table 66. H-RE Results Comparison (Phase II vs. Phase I).....	Pg. 130.
Table 67. Appearance of Repair Coatings.....	Pg. 131.
Table 68. Bend Adhesion Results for Repair Coatings.....	Pg. 132.
Table 69. Coating Thickness Results for Repair Coatings.....	Pg. 133.
Table 70. Unscribed Corrosion Resistance Results for Repair Coatings.....	Pg. 134.
Table 71. Scribed Corrosion Resistance Test Results for Repair Coatings.....	Pg. 136.
Table 72. Hydrogen Embrittlement/Reproducibility Test Results for the Repair Coatings.....	Pg. 138.
Table 73. Threshold Stress Intensities for SCC.....	Pg. 155.
Table 74. AMCOM Hydrogen Re-Embrittlement/Stress Corrosion Cracking Test.....	Pg. 160.

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Mr. Nathan Hughes from HAFB provided the military depot processed control cadmium and IVD-Al coated specimens. Mr. Luc Doan of Navy FRC-SW provided the IVD-Al coated panels for the Navy add-on portion of testing. Alternative coating providers included Dr. John Marshall at Marshall Laboratories, Mr. Kelly Donaldson of AlumiPlate Inc., Mr. Takeshi Kunihiro at Dipsol of America, Boeing Corporation (Seattle), and Boeing Corporation (St. Louis). Mr. Brian Placzankis of ARL in Aberdeen, MD performed some of the corrosion, embrittlement and fluid resistance testing while Mr. Paul Roser and Mr. Andy Schwartz of the Materials Division (AIR 4.3.4) at NAWCAD Patuxent River, Maryland also performed embrittlement and re-embrittlement testing. Westmoreland Mechanical Testing and Research performed the torque-tension and other fastener related tests.

Four major leveraged funding sources contributed to this project. ESTCP WP-0022 provided the major portion of funding for Phase I and Phase II coating test efforts, while the Naval Environmental Sustainment Development to Integration (NESDI) Project #408 provided funding for key Navy specific testing in Phase II including stress corrosion cracking, air and salt water fatigue, and cyclic salt spray (sulfur dioxide) corrosion testing in painted and unpainted, scribed and unscribed conditions. U.S. Air Force Pollution Prevention (P2) funds contributed to the test plan coordination and execution, AlumiPlate™ coating demonstration on F-35 landing gear as well as further scale-up of LHE Zn-Ni for landing gear applications through other contracted efforts. The Joint Group Pollution Prevention (JG-PP) provided some initial start-up funding for an LHE Zn-Ni coating demonstration at Navy FRC-SE although component demonstration was not completed during this effort due to factors discussed within the report.

EXECUTIVE SUMMARY

ESTCP project WP-0022 was initiated to provide an overarching and objective simultaneous evaluation of the most promising cadmium alternative coatings for high strength steel applications—prior to this project the leading coating alternatives had been evaluated in independent and limited scope studies where there were limited cross comparisons and inadequate scope of testing for DoD to make objective decisions. Project team stakeholders were essentially members of the Joint Cadmium Alternatives Team (JCAT) consisting of government and industry representatives who held interest in testing and implementing one or more of these alternatives. Coating performance was reviewed by JCAT team members during the course of the effort so that joint decisions were made with regard to which coatings would be dropped from further testing due to technical performance deficiencies. The JCAT made a consensus decision at the beginning of the project to test each cadmium alternative equivalently by each using a known hexavalent chromium (Cr(VI)) post-treatment process in order to avoid introducing two coating variables at once. One must maintain perspective that recent regulations have more strongly prioritized reduction of Cr(VI) so that alternative coating performance with non-Cr(VI) post-treatments is of relatively greater importance now. Some of this related work has been done by other organizations in parallel with the latter portion of WP-0022 so pertinent references will be mentioned here. A secondary objective of this project was to take maximum advantage of DoD depot coating processing and DoD laboratory test facilities where possible to ensure the coatings were applied, tested, rated and evaluated in a representative manner.

Along with the ESOH benefits of the cadmium alternative coatings being compared in this study there are process advantages and/or disadvantages for each. Essentially the electroplating based alternatives, Low Hydrogen Embrittlement (LHE) Zn-Ni (IZ-C17 process) and AlumiPlate™ coatings could offer similar component coverage as plated cadmium when using auxiliary anodes to coat internal recessed surfaces, while the sputtered aluminum process was specifically designed to provide sacrificial coating for larger internal diameters of U.S.A.F. landing gear due to the known limitation of Ion Vapor Deposited Aluminum (IVD-Al) to coating into recessed areas. No single alternative investigated here is yet authorized to replace cadmium in the varied cross-DoD applications on high strength steel although the outcome of this project at present includes one authorization letter along with significant technical data/guidance how and where these alternatives may be implemented with limited risk. Results from this project and a parallel U.S. Air Force demonstration scale-up effort have also recently resulted in a near-term path forward for LHE Zn-Ni implementation on some assets coated at Hill AFB / Ogden Air Logistics Center. When it comes to implementing these coatings on new platforms, such decisions are largely a collaborative OEM/Government function where component design and manufacturing considerations must be factored into component design decisions and each DoD acquisition entity is empowered to approve/disapprove these new coatings based on technical and field performance data.

Based largely on the favorable results of this project and extensive field experience with IVD-Al coatings NAVAIR prepared a limited authorization letter for the AlumiPlate™ coating on June 2009 as an equivalent or better performing alternative as compared to IVD-Al. The authorization limits the coating to applications specifically addressed within the letter and

requires the use of the highest performing corrosion resistant paint finishes in order to maintain an equivalent or better position relative to existing IVD-Al applications. For example the coating is not authorized for bulk unpainted applications like electrical connectors and fasteners at this time. The AlumiPlate™ process is domestically available in the U.S. but is currently sole-source and proprietary so that parts must be routed through the manufacturer, making OEM level implementation more feasible than military depot level. Although contained and managed at the manufacturing site the current process employs toluene and hazardous organometallic electrolytes, so the elimination of the cadmium by this method represents only one facet of cadmium reduction on HSS components. U.S. Air Force Pollution Prevention (P2) funds were used to initiate demonstration of AlumiPlate™ on F-35 aircraft landing gear through a contracted effort which began during Phase II of this test program.

The LHE Zn-Ni plating formulation developed to be more compatible with high strength steel substrates exhibited similar to better corrosion resistance than cadmium in many of the tests performed here and in the parallel U.S. Air Force effort. This is known as the IZ-C17 process, or LHE Zn-Ni (IZ-C17). The data generated in this ESTCP project still reflects some performance risk for in-service embrittlement (Phase I) and fatigue as observed in certain other Zn-Ni plating formulations, although these factors could be offset by performance gains in other key technical parameters. Plans for demonstration of LHE Zn-Ni at Navy FRC-SE had been initiated in 2008 through leveraged Joint Group on Pollution Prevention (JG-PP) funding although it was not fully engaged pending completion of all Phase II testing. Due to greater interest in LHE Zn-Ni by the Air Force, a parallel independent effort was launched to generate additional technical data on the coating plated from a demonstration size tank. This other data included such factors as process scale-up and bath maintenance issues, process chemistry stability by reformulating the LHE Zn-Ni (reformulation is identified as IZ-C17+), coating-substrate interactions at elevated temperatures, and greater emphasis on non-Cr(VI) post-treatments. Based on the interim results of this project, the parallel Air Force demonstration results as well as some platform specific issues, Hill AFB began the process of LHE Zn-Ni implementation in November 2010 via incorporation into appropriate Air Force documentation.

Sputtered aluminum coating implementation has also been led by Hill AFB personnel where it has been used for several years to provide internal diameter coating capabilities as an adjunct to their IVD-Al coating chambers, i.e. operated within the same vacuum chamber to apply more comprehensive coating coverage on components. These coatings have been utilized in fairly limited number of applications to date, and due to its cost it may be replaced by LHE Zn-Ni where engineering approval is obtained.

Each cadmium alternative coating system was evaluated against the JTP requirements and given pass/fail or semi-quantitative ratings depending on the performance parameter being tested. Electroplated Sn-Zn coating and Boeing's 'Acidic' Zn-Ni process were voted by JCAT to not continue into Phase II testing based on hydrogen embrittlement and re-embrittlement (H-RE) test results in Phase I. In both Phases I and II the AlumiPlate™ coating performed better than cadmium in a number of tests including key embrittlement, stress corrosion cracking, acidified (SO₂) salt fog, fluid corrosion resistance tests, etc. but did not perform quite as well in the long term salt fog corrosion tests when scribe damage was induced in the coating in accelerated neutral salt fog testing. LHE Zn-Ni performed well in most of the testing here and in particular

performed about the same as or better than cadmium in all but one of the accelerated corrosion tests (in galvanic assemblies tested in ASTM B 117 salt spray the LHE Zn-Ni coating developed higher resistance values than the other coatings). For LHE Zn-Ni, H-RE and fatigue remained the principal performance concerns especially for Navy applications. None of the alternatives evaluated matched cadmium plating with respect to torque-tension values after removing and replacing a fastener a number of times, however, this result reflects only one particular thread lubricant as specified in JTP. Torque-tension performance is important for applying known forces to fastened joints and both over- and under-torquing bolts could have adverse consequences.

ESTCP project WP-0022 ended after the Phase II testing was completed due to a number of factors. One was the lack of perceived need to test either sputtered aluminum or electroplated aluminum for air and corrosion fatigue performance given the similarity to IVD-Al (all commercially pure aluminum coatings) as shown in the Navy add-on testing. A second was the re-emphasis on testing and implementing cadmium alternative coating systems with non-Cr(VI) post-treatments to be in greater compliance with the changing global regulatory climate for cadmium and hexavalent chromium. Further repair coating characterization was also to have been a focus of Phase III.

For brush electroplated LHE cadmium replacements, the brush plated Zn-Ni formulation tested here performed best overall with the most favorable adhesion and corrosion resistance, although insufficient data was generated by the end of Phase II of this project for implementation decisions. The brush electroplated Sn-Zn repair coating exhibited concerns primarily with corrosion resistance and temperature limitation. The SermeTel[®] 249/273 sacrificial coating which may be applied with a paint brush or spray operation did not pass JTP bend adhesion and corrosion tests, however, it was the only repair alternative investigated here which suitable for operational level maintenance activities.

1.0 INTRODUCTION

This section provides a general overview of the most common environmental and industrial sources of cadmium exposure and the associated health risks followed by the more specific issue of cadmium alternatives for high-strength steel components such as landing gear. The objective of this project will be defined within this context followed by a more in-depth discussion of constantly evolving regulatory framework associated with the cadmium/hexavalent chromium coating which drove the initiation of this effort.

1.1 BACKGROUND

General Cadmium Alternatives Background

Cadmium coatings with hexavalent chromium (Cr+6 or Cr(VI)) post-treatments have been targeted for reduction by various environmental initiatives since the 1970's due to toxicity concerns with both cadmium and hexavalent chromium [1], and both are now known to be human carcinogens [2]. With respect to cadmium, the U.S. EPA Fact Sheet [3] lists various types of food as common sources of cadmium although the amount ingested is "about ten times less" than would cause kidney damage, the major target organ for ingested cadmium. Smokers are exposed to additional cadmium directly through cigarettes [2-4]. The major airborne sources of cadmium in the environment are listed as originating from fossil fuel and municipal waste burning power plants and metal smelting operations which process zinc, copper or lead [3]. If exposure is excessive these airborne sources can lead to lung and kidney diseases. Two recent studies have suggested that cadmium may also play a role in breast cancer [5] and it has also been more strongly correlated with the pulmonary diseases emphysema and chronic bronchitis [6]. While both studies require larger populations to be statistically valid they raise additional concerns about cadmium's role as a human health concern. It should be noted that the largest commercial use for cadmium by volume is nickel-cadmium (Ni-Cd) batteries (79%) overshadowing its use in pigments (11%) and coatings (7%), although minor amounts are included in stabilizers such as used for polyvinyl chloride (PVC) production, in cadmium-containing alloys for soldering/brazing and within electronic components [4].

Cadmium coatings have been employed extensively in the aerospace industry due to its unique functional and performance attributes in providing protection from corrosion on a wide variety of components including high strength steel fasteners such as bolts and pins to landing gear structural components where corrosion and in-service embrittlement protection have been key requirements for long term reliability and safety reasons. On fasteners cadmium provides some degree of lubricity where maintaining proper loading forces (torque-tension performance) is necessary, while in a rather different role it is also heavily used on aluminum shells of electrical connectors for maintaining electrical continuity, commonly described as an "olive drab finish". One of the occupational hazards of cadmium is that it's easily removed during paint stripping operations thereby contaminating the stripping media and creating large volumes of hazardous waste. Cadmium also tends to contaminate everything it physically contacts such as wiping rags, hydraulic oil, and masking materials [7].

Due to Environmental Safety and Occupational Health (ESOH) issues with vacuum deposited cadmium, IVD-Al was first implemented at some DoD installations in the late-1980's. There are a couple key limitations with the IVD-Al process including limited ability to coat internal and deeply recessed surfaces, and the coating does not pass re-embrittlement (also known as in-service embrittlement) tests per the High Strength Steel (HSS) Joint Test Protocol (JTP) Sec. 3.6.2 [8-9], however, it has been a preferred alternative to cadmium for higher temperature applications associated with engine components and also for select landing gear components due to cadmium's limitation to applications $\leq 450^{\circ}\text{F}$. It should be noted that application specific engineering considerations must always be factored in when determining the feasibility of a particular cadmium alternative (e.g., fundamental resistance to embrittlement phenomena, fatigue criticality, temperature excursions, potential for galling and/or galvanic corrosion with adjacent surfaces, the corrosion inhibiting performance of the final paint finish, etc.). Accordingly, it has been noted in more than one reference that no single alternative is likely to replace cadmium in its three primary DoD applications (high strength steel components, high strength steel fasteners and electrical connectors which are frequently 6061 aluminum). With this in mind, IVD-Al has been used with chromate (Cr(VI)) conversion coatings and chromate-inhibited primers thereby achieving the greatest total corrosion protection for the components. Some historical test data for IVD-Al in comparison to cadmium and other alternatives is presented in the summary section of the Society of Aerospace Engineer's report SAE AIR 5479 [7], "Environmentally Compliant Processes for Landing Gear". While IVD-Al does not perform exactly equivalent to cadmium in each JTP test, some are better while some are worse, it has functioned well enough in select applications to be used successfully for over 20 years.

ESTCP WP-0022 Project and Joint Cadmium Alternatives Team (JCAT) Background

For high-strength steel applications, a number of alternatives had been proposed to replace cadmium electroplating and replace or augment IVD-Al, however, additional testing was needed to validate whether the new coatings will meet all necessary DoD performance requirements. To this end, in 2002 the Air Force Research Laboratory (AFRL) contracted Concurrent Technologies Corporation (CTC), in cooperation with The Boeing Company (Boeing), to develop an appropriate test protocol. An initial test protocol had been prepared to delineate and describe the performance requirements for coatings that are applied to high strength structural alloy steel (>200 ksi) landing gear components as processed by Hill Air Force Base (HAFB)/Ogden Air Logistics Center (OO-ALC). CTC assisted Boeing in the establishment of the team, which included representatives from Boeing-St. Louis, Boeing-Mesa, Air Force Materiel Command (AFMC), AFRL, HAFB/OO-ALC, and CTC. Boeing designed an outline for the initial test protocol based on the performance requirements listed within the Cd plating specifications SAE AMS-QQ-P-416 and Military Standard (MIL-STD)-870B. These items were combined with input that had been gathered from the team and direct feedback from HAFB/OO-ALC personnel that focused on additional requirements not specifically called out within the specifications and current cadmium plating practices.

Per AFRL direction, this initial test protocol was then expanded to cover the Joint Services (Air Force, Army, and Navy/Marine Corps) as well as other OEMs to organize test requirements into both common and additional Service-specific needs. This reformed team kept the name JCAT and is generally managed by Navy personnel from Naval Air Systems Command (NAVAIR) at Patuxent River, MD. The JCAT team worked together to arrive at the test protocol released as

the “High-Strength Steel Joint Test Protocol (JTP) for Validation of Alternatives to Low Hydrogen Embrittlement Cadmium for High-Strength Steel Landing Gear and Component Applications—dated July 2003” [8], hereafter referred to as the JTP. The JTP was prepared to represent the minimum test procedures and performance requirements necessary for subject matter experts to consider implementation of cadmium alternative coatings on high risk, high-strength steel components such as landing gear.

In early 2004 CTC developed a corresponding JTP test execution plan which laid out a three-phase test approach per JCAT consensus whereby coating down-selection was planned at the conclusion of each phase. Phase I consisted essentially of screening tests to first eliminate any coatings which produced unacceptable process induced hydrogen embrittlement or in-service embrittlement of high-strength steels. Phase II was the most comprehensive test phase with the bulk of the common corrosion related and other performance testing included therein, with Phase III being largely focused on the more costly fatigue testing. Points of contact for the most recent ESTCP WP-0022 project participants including coating vendors, government test and evaluation laboratories and contractor participant are listed in Appendix A.

ESTCP WP-0022 Phase I Test Execution

Phase I tests were conducted from February 2005 through October 2005 at Patuxent River Naval Air Station and U.S. Army Research Laboratory (ARL). Phase I tests involved evaluation of all primary coatings in accordance with JTP Section 3.2.1 (Bend Adhesion), Section 3.6.1 (Hydrogen Embrittlement), Section 3.6.2 (Hydrogen Re-Embrittlement), and Section 6.1 (AMCOM In-Service Hydrogen Re-Embrittlement /Stress Corrosion Cracking), and evaluation of repair coatings per Section 3.2.1 (Bend Adhesion) and Section 3.6.1 (Hydrogen Embrittlement). Phase I test results were presented to the Joint Cadmium Alternatives Team (JCAT) during a WebEx™ telecon in November 2005 where a down-selection vote including all JCAT stakeholders was conducted. Electroplated Tin-Zinc was dropped from further consideration due to poor hydrogen embrittlement performance while Boeing Seattle's Electroplated Acidic Zinc-Nickel was dropped due to its susceptibility to hydrogen re-embrittlement. All repair coatings were selected to continue through Phase II testing. Phase I test results were published in a NAWCAD Technical Report, NAWCADPAX/TR-2006/164 [9].

ESTCP WP-0022 Phase II Test Execution

The ESTCP funded portion of the WP-0022 Phase II testing was coordinated through CTC via U.S. Air Force contract number FA8601-05-F-0011 while the NESDI funded U.S. Navy specific testing was coordinated and performed by NAVAIR Patuxent River. CTC was tasked with being the focal point for collection and distribution of test materials and coated coupons for this project, performing some of the tests and developing interim and final test briefings and reports up to and including the Phase II Joint Test Report (JTR). Boeing Seattle participated as a subcontractor to CTC to provide consulting services to aid in interpretation and discussion of test results. Phase II was conducted over the period December 2006 through June 2009 according to the test execution plan developed by JCAT [10].

Coatings downselected for inclusion in Phase II had been previously established by Joint Cadmium Alternatives Team (JCAT) stakeholders through a voting process. Primary alternative coatings evaluated in the Phase II effort were limited to magnetron sputtered aluminum,

electroplated aluminum and LHE Zn-Ni (IZ-C17 process) which were compared against both control coatings LHE Cd and IVD aluminum. Repair coatings that were evaluated included brush electroplated Zn-Ni, brush plated Sn-Zn and spray/brush-applied SermeTel[®] 249/273. The control coating for repair was brush plated LHE Cd. Testing was performed by NAVAIR Patuxent River, MD, U.S. Army Research Laboratory, Aberdeen Proving Grounds, MD, Westmoreland Mechanical Testing and Research (WMTR), and Concurrent Technologies Corporation (CTC), Johnstown, PA). Phase II tests were conducted IAW the “High Strength Steel Joint Test Protocol (JTP) for Validation of Alternatives to Low Hydrogen Embrittlement Cadmium for High Strength Steel Landing Gear and Component Applications.”

Interim results for ESTCP WP-0022 were presented and/or published at several technical meetings in the 2005-2009 timeframe as outlined in Table 1, while the final WP-0022 JTR was not released until May 2010 [11]. Also included in this table are a couple noteworthy references which emerged during this timeframe regarding cadmium alternative implementation guidance. The most recent guidance on environmentally compliant finishes specific to high strength steel landing gear was published in 2007 as SAE AIR 5479 [7] which is valuable in that it covers considerations beyond just cadmium such as the inclusion of non-Cr(VI) surface treatments, non-Cr(VI) paint systems and hard chromium (Cr(VI) electroplating) alternatives as a full scope protective coating system for components. The second was a Battelle Laboratories study funded through the U.S. Air Force which semi-quantitatively ranked the most promising cadmium alternatives according to various DoD application specific requirements including high strength steel components, fasteners and electrical connectors [12].

It was mutually agreed to conclude ESTCP WP-0022 after the Phase II effort due partially to the lack of a perceived need to test either sputtered aluminum or electroplated aluminum for additional fatigue characterization given the similarity to IVD-Al shown in the Navy specific testing phase of the effort (all are commercially pure aluminum coatings albeit with somewhat different physical/microstructural properties), and the re-emphasis on validating cadmium alternatives coupled with non-Cr(VI) post-treatments.

**Table 1. Timeline for Cadmium Alternatives Testing and Guidance Documentation
(Relevant to High Strength Steel Applications per ESTCP WP-0022)**

YEAR	Significant Event Related to Project WP-0022
1987	IVD aluminum coatings per MIL-DTL-83488 (ion-vapor deposited aluminum, or IVD-Al) were selectively applied to some high-strength steel components at U.S. military repair facilities.
2002	Analysis of Alternatives (AoA) for WP-0022 test coatings, Screening of background information and alternatives.
2003 (July)	WP-0022 JTP was issued, “High Strength Steel Joint Test Protocol for Validation of Alternatives to Low Hydrogen Embrittlement Cadmium For High Strength Steel Landing Gear and Component Applications”. Prepared by The Boeing Phantom Works, Seattle WA and Concurrent Technologies Corporation (Contract#CTC/LAU-CL2402-02) for AFRL Task Order 5TS5702D035M.
2004 (Mar.)	Project Test Plan developed by CTC through contract with AFRL, GSA TO# 5TS5702D035H [10].
2005 (Nov.)	ESTCP WP-0022 Phase I Preliminary Results Available - JCAT reviewed Phase I embrittlement results and voted to drop two primary coatings from consideration and retain all repair candidates.
2006	Semi-quantitative guidance rankings were generated for the most promising cadmium alternatives in each of the key functional areas: landing gear, fasteners, and electrical connectors. U.S. Air Force ASC/ENVV report from Battelle Laboratories: Pollution Prevention Technology Integration Support Contract, FY06 Contract Deliverable, DAAD19-02-D-0001/DO0869/TCN06131 [11].
2007 (Jan.)	ESTCP WP-0022 Phase I JTR was issued for this project via NAWCAD Technical Report – NAWCADPAX/TR-2006/164 [9].
2007 (July)	Preliminary results from ESTCP WP-0022 were mentioned in SAE Aerospace Information Report AIR 5479A “Environmentally Compliant Finishes for Landing Gear”. Contains a summary of environmentally compliant finish testing for landing gear which is not limited to cadmium as the only Hazmat (includes Cr(VI) ‘Hard Chrome’ plating and hexavalent chrome paint finish alternative categories) [7].
2007 (Dec.)	Navy funded portions of WP-0022 test results were presented by NAVAIR at NACE Tri-Services meeting in Denver, CO, Dec. 2007 and also published in the proceedings as paper P1792 [12]. Status briefing for ESTCP WP-0022 was provided by AFRL.
2008 (Feb.)	WP-0022 Status Briefing was presented at ASETS Defense Conference, New Orleans LA.
2009 (Nov.); 5 April 2010 Public Release Authorized	WP-0022 Phase II JTR was issued for this project – Weapon System Technical, Logistical, and Sustainment Support FA8601-05F-0011, Subtask 024 (CDRL A0020), Testing Cadmium Alternatives for High Strength Steel Phase II Joint Test Report [13]. Air Force public release Case Number 88ABW-2010-1828.

1.2 OBJECTIVE OF THE PROJECT

ESTCP project WP-0022 was initiated to provide an objective evaluation of the most promising cadmium alternative coatings for DoD high-strength steel applications—prior to this project the leading coating alternatives had been evaluated in independent studies where there were incomplete cross comparisons and insufficient scope of testing for DoD to make objective decisions. Project team stakeholders were essentially members of the Joint Cadmium Alternatives Team (JCAT) consisting of government and industry representatives who held interest in testing and implementing one or more of these alternatives. Coating performance was reviewed by JCAT team members during the course of the effort so that joint decisions were made with regard to which coatings would be dropped from further testing due to technical performance deficiencies. The JCAT made a consensus decision at the beginning of the project to test each cadmium alternative equivalently by each using a known hexavalent chromium (Cr(VI)) post-treatment process in order to avoid introducing two coating variables at once. Regulatory drivers for this project have changed during the execution of this project, so one must maintain perspective when reviewing this report that recent regulations have more strongly prioritized reduction of Cr(VI); hence, alternative coating performance with non-Cr(VI) post-treatments is of relatively greater importance now. Some of this related work has been done by other organizations in response to these changing drivers in parallel with the latter portion of WP-0022; pertinent references will be mentioned here.

A secondary objective of this project was to take maximum advantage of DoD depot coating processes and DoD laboratory test facilities where possible to ensure the coatings were applied, tested and ranked in a representative manner. Coating performance was reviewed by JCAT team members at various status briefings during the course of this project to keep stakeholders information and so that joint decisions could be made with regard to which coatings would continue testing and which would be dropped from consideration (principally done after Phase I).

JCAT stakeholders determined that the most promising primary alternative coatings to be evaluated in the Phase I effort were to include Magnetron Sputtered Aluminum “Sputtered Al”, Electroplated Aluminum (AlumiPlate™ process), Low Hydrogen Embrittlement (LHE) Electroplated Zinc-Nickel (IZ-C17 process), Electroplated “Boeing Acidic Zinc-Nickel” and Electroplated Tin-Zinc. Control coatings were LHE Electroplated Cadmium and Ion Vapor Deposited Aluminum (IVD-Al). Several repair coatings were also evaluated, including Brush Electroplated Zinc-Nickel (SIFCO 4018), Brush Electroplated Tin-Zinc (LDC 5030), and Spray/Brush-applied SermeTel® 249/273. The control coating for repair evaluation was Brush Electroplated LHE Cadmium (SIFCO 2023). Table 2 provides the basic information regarding each of these coatings, including the coating vendor/provider for each alternative and to what specification each was applied as well as representative coating composition ranges. More detailed process flows and procedures are provided in Appendix B. Figures 1 and 2 illustrate the appearance of the coated primary and repair test coupons, respectively, prior to testing. Shown are 1x4 inch bend adhesion test panels, C-rings for accelerated in-service embrittlement testing, and notched round bars for both process hydrogen embrittlement testing in air and re-embrittlement (in-service embrittlement) immersed in saltwater and other environments.

Table 2. Coating Information for Each Cadmium Alternative Tested
(See Appendix B for Detailed Process Flows)

Coating	Coater	Coating Process Specification or Product	Post-Plate Hydrogen Relief Bake	Representative Coating Thickness	Other
LHE Cadmium	Hill Air Force Base, Utah	MIL-STD-870	Yes	Targeted 0.6 mil	
IVD Aluminum (IVD-Al)	Hill Air Force Base, Utah	MIL-DTL-83488 Class 2	No	Targeted minimum 0.5 mil	Unpeened
Sputtered Aluminum	Marshall Laboratories, Boulder CO	Marshall Lab Process	No	Targeted 0.5 to 0.8 mil	Conversion coating applied at NAVAIR (Ph. I), CTC (Ph. II)
Electroplated Aluminum, (AlumiPlate™)	AlumiPlate, Incorporated, Minneapolis, MN	MIL-DTL-83488 Class 2	No	Targeted 0.9 mil	
LHE Zinc-Nickel	Dipsol of America, MI	Boeing/Dipsol Procedure IZ-C17	Yes	Targeted 0.5 mil	84-88% Zn 12-16% Ni
Acidic Zinc-Nickel	Boeing Seattle, WA	BAC 5637	Yes	Targeted 0.5 mil	90.5-91.5% Zn 8.5-9.5% Ni
Tin-Zinc	Dipsol of America, MI	AMS 2434	Yes	Targeted 0.5 mil	75-85% Sn 15-25% Zn
Repair Coatings					
Brush Cadmium	Boeing St. Louis, MO	SIFCO 2023	No	Targeted 0.5 mil	
Brush Zinc-Nickel	Boeing St. Louis, MO	SIFCO 4018	No	Targeted 0.5 mil	88-92% Zn 8-12% Ni
Brush Tin-Zinc	Boeing St. Louis, MO	LDC 5030	No	Targeted 0.5 mil	70-75% Sn 25-30% Zn
SermeTel® 249/273	Boeing St. Louis, MO	Sermetech Engineering Bulletin 249	No	Targeted 0.5 mil	Constituents: Al and Zn
Note: All specimens received a subsequent chromate-based conversion coating. All coaters were asked to target 0.5 mil coating thickness in both phases of ESTCP WP-0022. Detailed process flows are listed in Appendix B, including substrate preparation and other pre-treatments, process conditions, and post-treatments.					

Depending on the technical performance of each alternative evaluated, it was understood that OEMs and DoD repair facilities would consider implementing one or more of these alternatives to cadmium plating where engineering approval was obtained. The JTP included some additional qualification tests important to OEMs which were not reflected in this funded effort, such as coating impact resistance, additional quality control tests for H-E and H-RE performance and additional fatigue tests with different load ratios, specimen geometries, etc.

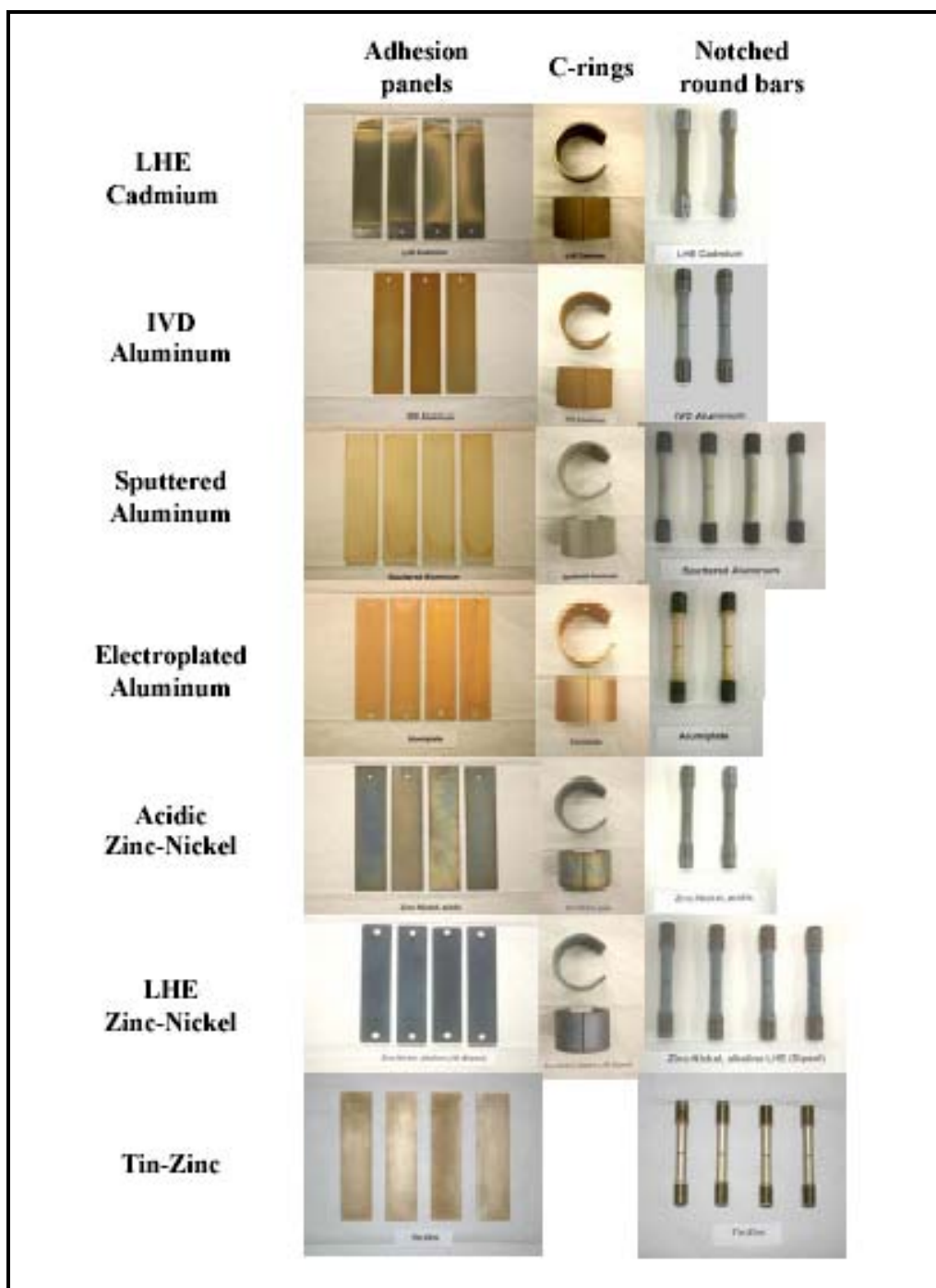


Figure 1. Coated Test Coupons Prior To Test – Primary Coatings

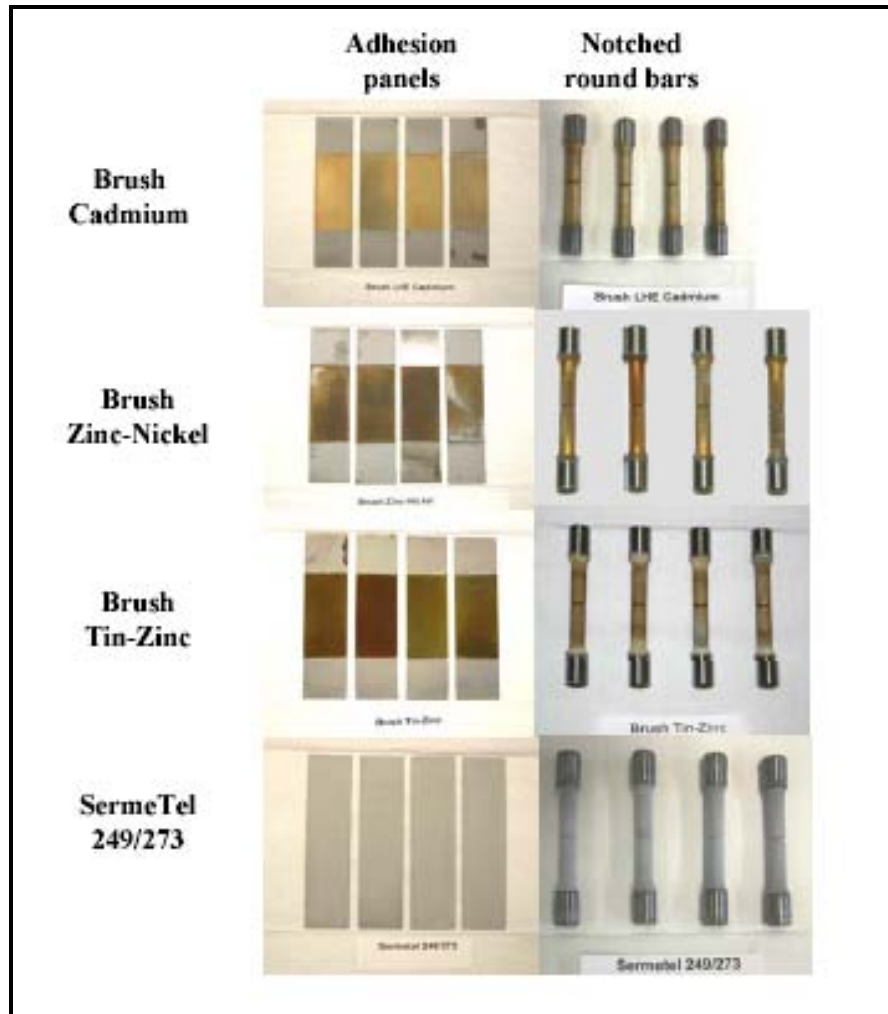


Figure 2. Coated Test Coupons Prior To Test – Repair Coatings

1.3 REGULATORY DRIVERS

There are significant domestic and foreign regulatory drivers to reduce cadmium (Cd) and its proven legacy post-treatment hexavalent chromium (Cr(VI)). Domestic regulations were promulgated first beginning in the 1940's by occupational safety organizations like ANSI, while later numerous other regulations were added to the list such as OSHA and NIOSH, the Clean Water Act and Clean Air Act, more than one Executive Order, and recent DoD Directives. There are also local guidelines which may sometimes be a function of needs and capabilities of the local waste water treatment authority. The most overshadowing foreign drivers to date have been the European Reduction of Hazardous Substances (RoHS) and Regulation, Evaluation, Authorisation and Restriction of Chemicals (REACH) legislation which strongly influence the global commercial marketplace which overshadows the specific needs of the DoD. Other non-European countries have also been enacting their own guidelines to protect their citizens and environment making the regulatory landscape more difficult to navigate.

Brief U.S. Regulatory History for Cadmium and Hexavalent Chromium

The regulatory timeline for cadmium may be summed up by this excerpt from OSHA's website [1]: "OSHA's existing permissible exposure limits (PEL) for cadmium were originally developed by the American National Standards Institute. In 1941 the American Standards Association (now American National Standards Institute, or ANSI) set as guidelines an American Defense Emergency Standard of 1000 $\mu\text{g}/\text{m}^3$ for cadmium and its compounds. This was done to reduce discomfort from exposures to cadmium and to reduce the incidence of acute health effects. ANSI revised its standard to current levels (ANSI Z37.5, 1970) which OSHA adopted in 1971 as a national consensus standard under section 6(a) of the Occupational Safety and Health Act of 1970 (29 U.S.C. 655). These PELs, as specified in 29 CFR 1910.1000, Table Z-2 are an 8-hour time-weighted average (TWA PEL) of 100 $\mu\text{g}/\text{m}^3$ for cadmium fume with a ceiling concentration of 300 $\mu\text{g}/\text{m}^3$ and an 8-hour TWA of 200 $\mu\text{g}/\text{m}^3$ for cadmium dust with a ceiling concentration of 600 $\mu\text{g}/\text{m}^3$. OSHA's existing PEL in the construction industry is 100 $\mu\text{g}/\text{m}^3$ for cadmium oxide fumes and 200 $\mu\text{g}/\text{m}^3$ for metal dust and soluble salts (29 CFR 1926.55)."

The 1992 OSHA Expanded Standard for Cadmium reduced the permissible exposure limit (PEL) for airborne cadmium to 5 $\mu\text{g}/\text{m}^3$ which reduced the Action Level (AL) to 2.5 $\mu\text{g}/\text{m}^3$. This standard went into effect in 1996 and placed many manufacturing and overhaul operations at risk of violating AL and/or PEL. This standard put in place more stringent overhaul process control, worker medical surveillance and record keeping. Worker medical surveillance programs affect some, but not all, DoD overhaul facilities dependent on measured exposure levels. These ESOH programs effectively increase the life-cycle cost of continuing to use Cd. Cadmium levels in effluent are regulated by the Clean Water Act, but the needs and capabilities of the local waste water treatment authority require their involvement in establishing individual discharge standards.

A significant contribution to cadmium's corrosion protective value comes from the post-treatment process (historically Cr(VI), hexavalent chromium) and the OSHA Cr(VI) guidance issued on 28 February 2006 lowered the PEL for worker exposure to Cr(VI) to 5 $\mu\text{g}/\text{m}^3$ from its previous 52 $\mu\text{g}/\text{m}^3$ (based on 8-hour time-weighted averages) [1]. *Thus there is environmental pressure on both cadmium and its commonly used hexavalent chromium post-treatment.* A certain amount of relief was provided to larger facilities representative of the aviation industry which could not economically meet these Cr(VI) limits in the entire facility, but it was still required to protect workers by making greater use of personal protective equipment (PPE). Most recently, the Deputy Undersecretary of Defense Acquisition Technology and Logistics (AT&L) issued a memorandum on 8 April 2009 "Minimizing the Use of Hexavalent Chromium (Cr(VI))" which directed more aggressive action to reducing hexavalent chromium usage while acknowledging that this reduction effort must be balanced with life-cycle performance (i.e. corrosion resistance) [14]. This has placed additional incentive upon developing safe and cost-effective alternatives to the cadmium/Cr(VI) coating system.

In March 2007 the U.S. EPA released its exposure guidance document "Framework for Metals Risk Assessment" which has some bearing on how the toxicity of Cd and chromium are assessed [15]. This document made it policy that the toxic properties of metals and the associated risks be evaluated on more specific scientific data for each metal, the forms of the metals or metal compounds, and the ability of the target organism to regulate, process and/or store the

metal. Regulatory limits for metals/compounds can now be more appropriately focused on those forms of metals or metal compounds that may pose potential hazards rather than assigning a risk assessment to a single metal or more general classes of metal compounds. General prohibition of metals/materials creates confusion on what real hazard levels actually exist. For example, some nickel compounds may be considered toxic, while others are not. Similarly, hexavalent chromium liquids or compounds pose risks that chromium metal (think ‘chrome’ motorcycle parts) or other chromium compounds do not (e.g., Cr(III)). Trivalent chromium (Cr(III)) is, in fact, an essential nutrient. For cadmium, the EPA framework means that different limits for cadmium in food and drinking water have been derived based on the rationale that the bioavailability of cadmium in water is greater than that of cadmium in food by a factor of 2 [15].

Recent changes in toxicology concerns about cadmium have also prompted DoD’s MERIT Directorate (Materials of Evolving Regulatory Interest Team) to initiate a new assessment for cadmium (2009). Cadmium has historically been associated with chronic kidney disease and cancer, however, new data from National Institute of Environmental Health Sciences (NIEHS) has strengthened cadmium’s link to breast cancer. As a carcinogen, Cd targets several sites that are considered endocrine-sensitive, and some new data suggest that breast cancer may be among them although further studies are necessary [5]. Suffice it to say that the regulatory climate is ever evolving as new data becomes available and that continual attention must be paid to global, federal, state and local ESOH driving forces when it comes to materials replacement efforts. This project specifically addressed only the cadmium component of the cadmium/Cr(VI) coating system environmental issues as described above although the alternate coatings investigated here all have some degree of compatibility with non-hexavalent chromium post-treatments.

2.0 TECHNOLOGY DESCRIPTION

2.1 CADMIUM AND ALTERNATIVE COATING TECHNOLOGIES

There are two primary types of processes that may be used for deposition of cadmium and its alternatives in this study—electroplating and vacuum deposition. Cadmium may be deposited either by electroplating (SAE AMS QQ-P-416, MIL-STD-870, AMS 2401) or vacuum deposition (MIL-C-8837, SAE AMS 2426). Typically cadmium coatings are specified using a hexavalent chromium (Cr(VI)) rinse or post-treatment using a ‘type’ designation in the specification (e.g., MIL-STD-870 Type II). The MIL-C-8837 vacuum cadmium specification requires the application of chromated post-treatment unless otherwise specified. The electroplating process for depositing Cd coatings is similar to any other electroplating process in that it requires a chemical bath containing dissolved metal ions, in this case cadmium salt(s) which have historically been cyanide salts plus other constituents to increase conductivity of the bath and maintain proper acidity/alkalinity, along with the means to apply electrical current to parts. Cadmium metal ions are thereby reduced into metallic coatings on the parts by the electrical current. Figure 3 shows a typical commercial electroplating operation.

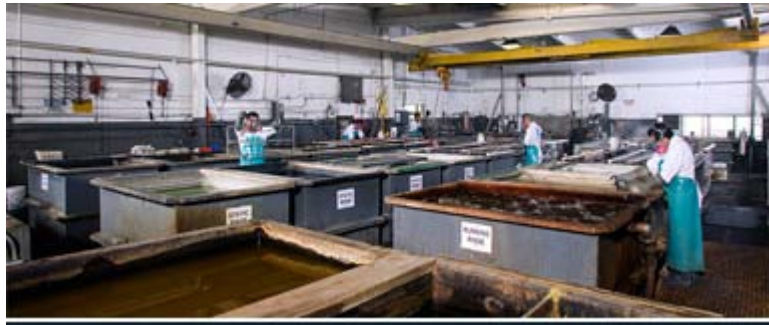


Figure 3. Typical industrial electroplating operation (Source: www.embee.com)

By contrast, vacuum deposition systems typically utilize large cylindrical vacuum chambers as shown in Figure 4. Vacuum cadmium coatings have typically been used for the highest strength steel components, i.e. those at greatest risk of becoming environmentally embrittled when placed into harsh service environments. Corrosion mechanisms generate hydrogen which can cause substantial reduction in mechanical properties.



Figure 4. Typical commercial vacuum coating deposition chamber (Source: www.embee.com)

In this project, commercially pure aluminum coatings were deposited using three distinctly different methods as further described below (IVD-Al, magnetron sputtered aluminum, and electroplated aluminum (AlumiPlate™)) all of which would meet the requirements of MIL-DTL-83488, the high purity aluminum coating specification. There is a separate AMS specification which is specific to the IVD-Al coating (AMS 2427). Both IVD-Al and sputtered aluminum coatings involve vacuum based deposition processes, while the AlumiPlate™ process is a unique non-aqueous electroplating technology (solution is not water based). Low hydrogen embrittlement (LHE) alkaline Zn-Ni (IZ-C17) is deposited by an aqueous based electroplating method most similar to electroplated cadmium as represented in Figure 3. The following subsections will further describe these alternative coating processes.

2.1.1 Ion Vapor Deposited Aluminum (IVD-Al) Process

The IVD aluminum coating process is performed in a vacuum chamber such as shown in Figure 5. The IVD-Al coating is specifically described by the AMS 2427 specification, but it also meets the requirements of MIL-DTL-83488—the generic high-purity aluminum coating specification—which permits either presence or absence of a post-treatment. In MIL-DTL-83488, Type II indicates the coating is chromated while the AMS 2427 specification references AMS 2473 which is a performance based conversion coating specification for aluminum. Corrosion protection is afforded to over 900°F with these high purity aluminum coatings, which represents one significant benefit over cadmium. Cadmium metal is limited to 450°F applications because it can cause embrittlement of high-strength steels at elevated temperatures.



Figure 5. Vacuum Coating System at Hill Air Force Base / Ogden Air Logistics Center
(Source: www.hazmat-alternatives.com)

Corrosion resistance of commercially pure coatings is listed in the specifications as a function of coating thickness while MIL-DTL-83488 also provides corrosion resistance quality control guidelines as a function of the presence or absence of a Cr(VI) post-treatment. The AMS 2427C

specification also notes that the corrosion resistance and adhesion of the IVD-Al coating is dependent on proper control of the glass or ceramic bead blasting “peening” process which is used to densify the coating by closing up the as-deposited interconnected porosity. If not aggressive enough the peening process can allow the coating to retain interconnected porosity, but if too aggressive the peening process can lead to thin and/or disbonded coatings. The IVD-Al process is performed in a vacuum chamber which has been backfilled with inert gas (argon) which enables parts to be subjected to a glow discharge or sputter cleaning operation to improve adhesion of the aluminum coating. Heated evaporator boats in the chamber are continuously fed with 1100 grade (99% minimum purity) aluminum wire which evaporates and transfers to the component(s). Depending on orientation of part surfaces in the chamber, IVD-Al coating thickness may not be equivalent on all areas of the coated part especially for internal diameters which could receive little or no coating coverage, although over years of implementation the process has evolved substantially to minimize numerous issues experienced earlier (see Sec. 2.2). Large components may be physically restricted from IVD-Al coating by the dimensions of the vacuum vessel. Testing in this and other efforts have shown the IVD-Al coating to be more protective than Cd in certain types of corrosion tests, while less protective in other tests.

2.1.2 Magnetron Sputtered Aluminum Process

Within the context of this effort, the magnetron sputtered aluminum process had been specifically developed to coat internal diameter (ID) surfaces which the baseline IVD-Al process does not adequately coat, so it may be considered as a standalone method to coat surfaces but more importantly as an incremental improvement to the IVD-Al deposition process.



Figure 6. Example of Industrial Sputter Coating Apparatus (Source: www.engineering.indiabizclub.com)

As an adjunct to the IVD-Al process an existing IVD chamber (such as shown in Figure 5) would be retrofitted with a sputter capability by design of appropriate sputter target hardware which may be specific to given geometries / size ranges of components to be internally coated. Figure 6 shows an example of an industrial sputter coating unit used for coating commercial components.

2.1.3 Electroplated Aluminum Process (AlumiPlate™ Coating)

Electroplated aluminum is commercially produced via the Siemens Sigal® process which was developed by the German company Siemens AG over the course of 30 years and most recently processed in Europe at Aluminal Corporation. The process was licensed in the United States to AlumiPlate, Inc. in 1995. The coating is deposited by electrodeposition similar to Cd although the bath is inherently non-aqueous and has special handling requirements explained further below (electroplating aluminum coatings from aqueous (water-based) solutions is not electrochemically possible). One advantage this coating process offers over IVD-Al is the ability to coat internal diameters by using auxiliary anodes similar to conventional electroplating techniques. Earlier data had suggested that the coating corrosion resistance and density were superior to IVD-Al although both comply to the MIL-DTL-83488 high-purity aluminum specification. Part size and geometry may be limited by existing tank dimensions and potentially by overhead crane carrying capacity. Compared to the vacuum based processes described above, the AlumiPlate™ process carries the additional process ESOH issues of being flammable (toluene-based) and having pyrophoric alkylaluminum constituent components.



Figure 7. AlumiPlate™ Coating Process Line
(Source: www.hazmat-alternatives.com)

Since conventional aqueous plating of aluminum is not electrochemically possible, attempts to improve upon the Siemens Sigal[®] process to mitigate these less desirable process characteristics will still likely involve some type of non-aqueous plating chemistry and a controlled environment.

2.1.4 Zinc-Nickel Electroplating

Various zinc-nickel plating bath chemistries have been developed over the past several decades ranging from acidic to alkaline bath pH values. These plating baths are capable of depositing a wide range of Zn-Ni coating compositions which has a large effect on coating mechanical/physical properties and functional properties such as corrosion resistance. Early versions of zinc-nickel plating were not suitable for high-strength steel applications. There are two primary coating specifications which narrow the coating composition to specific ranges, both of which permit either acid or alkaline formulations. AMS 2417G permits a wider range of nickel contents (6-20% Ni) than does ASTM B 841 (5-12%). Two different zinc-nickel plating formulations were tested in this project. One formulation which deposits ~9% nickel is referred to in this report as 'Acidic Zn-Ni (Boeing)' electroplating process. This formulation is deposited IAW Boeing specification BAC 5637. With respect to high-strength steel substrate compatibility, the latest alkaline LHE Zn-Ni plating formulation has demonstrated a favorable balance of coating properties. Both processes offer the potential to be a drop-in replacement for cadmium electroplating in existing infrastructure (Fig. 8). The alkaline formation tested in this project is known as the IZ-C17 process, and it deposits coatings with higher nickel content than the acidic Zn-Ni process. Both processes will be described in greater detail in the next section (Sec. 2.2, Technology Development).



**Figure 8. Low Hydrogen Embrittlement (LHE) Zinc-Nickel Plating, IZ-C17 Process
(Source: ES3 Inc.)**

2.1.5 Tin-Zinc Electroplating Process

The tin-zinc plating process tested in this effort per JCAT recommendation was developed by Dipsol of America, Inc., and is referred to as product designation SZ-240. The coating is electroplated out of a slightly acidic bath and results in a tin-zinc composition containing from 15-25% zinc. The process is conducted using infrastructure similar to the other aqueous based plating processes being investigated (zinc-nickel formulations) as shown in Figure 8.

2.1.6 Brush Cadmium Repair Coating Alternatives

While identifying a primary coating capable of replacing Cd and/or IVD-Al was the main goal of this project, identifying a coating capable of replacing brush plated cadmium for touch up and repair of the sacrificial coating is also important. The phrases “brush plating” and “selective electrodeposition” are used interchangeably, and AMS 2451 and MIL-STD-865 cover general coating requirements for selective electrodeposition. Repair of damaged cadmium coatings on landing gear and other high strength steel surfaces used to require disassembly, reapplication of cadmium electroplating, and then a baking operation at 190°C (375°F) for between 4-24 hours to alleviate hydrogen embrittlement concerns. A low hydrogen embrittlement cadmium brush plating solution was developed in the late 1960’s which did not require a bake after application, and therefore no expensive and time consuming disassembly of the component, industries took notice. Numerous aircraft and landing gear manufacturers began adding the LHE brush cadmium plating process to their specifications. The original LHE cadmium plating process is a registered trademark of Selectrons Ltd., which was acquired by SIFCO Industries Inc. in 1992.

The following repair coatings were selected by JCAT for evaluation in both Phases I and II of this project: Brush plated Zn-Ni (SIFCO 4018), brush plated Sn-Zn (LDC 5030), and a sprayed Al-ceramic (SermeTel[®] 249/273). Brush plated LHE cadmium (SIFCO 2023) was utilized as the baseline repair coating. While repair coatings are typically used to deposit a protective layer on areas where the primary coating has been damaged or compromised, ESTCP Phase II testing focused on evaluating the performance of repair coatings that have been deposited on bare substrates, in accordance with the JTP.

Two of the repair coatings tested in this project were applied by brush plating methods. Figure 9(a) illustrates an industrial application for mechanically operated selective plating, however, Figure 9(b) better represents the manual application of either brush Sn-Zn or brush Zn-Ni to test coupons in this project. The SermeTel[®] 249 coating may be applied using a paint brush but can also be thinned and sprayed like a spray paint. The SermeTel[®] 249 is cured via the application of SermeTel[®] 273, making it the most viable field-repair option as it is not dependent on an electrical rectifier for coating application.



Figure 9. (a) Industrial selective plating operation (Source: www.sifcoasc.com); (b) Application of a brush plated Zn-Ni formulation (Source: Navy FRC-SE)

2.2 TECHNOLOGY DEVELOPMENT

2.2.1 Ion Vapor Deposited Aluminum (IVD-Al) Technology Development

The Ion Vapor Deposited Aluminum (IVD-Al) process was developed in the 1960s by McDonnell Aircraft Company (now a part of Boeing Corporation) as a replacement for cadmium coating processes. Full scale production feasibility of IVD-Al was first demonstrated at Naval Air Rework Facility San Diego in 1974. The standard process included a rack from which parts are suspended or affixed, and fixed evaporator sources positioned down the center of the coater. For coating small parts, a barrel coater was developed which originally coated 75 pounds of steel fasteners during a coating run. By the early 1980s, improvements had been made which included moving evaporator sources and the capability to rotate part racks/fixtures both of which promoted more even coating deposition. Applications that have been reported include high-strength steel parts such as landing gear, fuel and pneumatic line fittings (in place of anodizing), electrical connectors, fasteners, electrical black boxes, corrosion and SCC protection of depleted uranium and titanium alloys, and electromagnetic interference compatibility [16]. The coating process became fairly widely implemented in U. S. military depots by the late 1980's due to ESOH issues with not just cadmium electroplating but also the vacuum cadmium coating process (MIL-C-8837). By that time, coating chambers were available with the capability to rotate parts to produce more evenly coated surfaces. The IVD-Al coating has reportedly been effective in a fairly wide variety of applications up to and including landing gear sized components [16-19]. The coating has historically been used in U. S. military landing gear applications with chromate post-treatments as well as chromate inhibited high-solids (solvent-based) epoxy primers to assure the best corrosion protection in operational environments, and is currently used on ~90% of high strength steel parts at Navy FRC-SE today.

2.2.2 Magnetron Sputtered Aluminum Process Technology Development

Sputtering processes developed very rapidly in the 1970s to primarily serve the semiconductor industry which is dependent on sputtering for mass production. The sputtering process was not tailored for landing gear components until the U.S.A.F. led these efforts in the 1990's. Boeing

first demonstrated the sputtered aluminum process which was also called “Plug & Coat” on internal surfaces of U.S. Air Force landing gear around 1999 [20].

2.2.3 Electroplated Aluminum (AlumiPlate™ Coating) Process Technology Development

Electroplated aluminum is commercially produced via the Siemens Sigal® process which was developed by the German company Siemens AG over the course of 30 years and licensed in 1995 in the United States to AlumiPlate, Inc. In Europe, the company Aluminal GmbH & Co. was the manufacturer for the electroplated aluminum coating although it ceased operations in August 2009, eliminating one source for electroplated aluminum. This process uses a flammable solution (toluene-based) and employs pyrophoric alkylaluminum constituent components and is therefore operated in an environmentally controlled process line where oxygen and humidity have upper control limits. Since conventional aqueous plating of aluminum is not electrochemically possible, attempts to re-engineer the Siemens Sigal® process to mitigate these negative process characteristics will still involve some type of non-aqueous plating bath and most probably a carefully controlled environment.

There have been some DoD Small Business Innovative Research (SBIR) topics released related to discovering and developing less process stringent methods to deposit dense pure aluminum coatings including more environmentally preferred solvents for this process or novel ionic liquids.

2.2.4 Acidic Zinc-Nickel (Boeing) Electroplating Technology Development

In the late 1960's and 1970's researchers were actively interested in Zn-Ni electroplating as an alternative to cadmium for corrosion protection of commercial steel products [21,22]. By the 1980's researchers were beginning to focus on a particular range of plated Zn-Ni coating chemistries and crystal structure which optimized corrosion resistance. Nounie et. al. [23] determined that single phase gamma Zn-Ni (γ -Zn-Ni) offered the greatest corrosion resistance, while others contributed to the understanding of the range of nickel content which would be most compatible with high strength steel substrates [24,25].

Boeing developed their acidic zinc-nickel electroplating process in the 1980's and performed scaled-up demonstrations beginning in 1983.

2.2.5 LHE Zinc-Nickel (IZ-C17 Process) Technology Development

The formulation of LHE Zn-Ni tested in this project was co-developed by Boeing Corporation and Dipsol of America. They developed a modified plating chemistry which was more compatible with high strength steel alloys (i.e., less embrittling than the standard chemistry) while maintaining an optimized range of plated γ -Zn-Ni coating. The process chemistry underwent slight optimization by a U.S. Air Force led team over the last couple years during this project but is not believed to influence the results herein. The chemistry modification resulted in the process name being changed from IZ-C17 to IZ-C17+ and results in this report are based on the IZ-C17 process.

2.2.6 Tin-Zinc Electroplating Process Development

The tin-zinc plating process tested in this effort was developed by Dipsol of America, Inc., and is referred to as product designation SZ-240. The coating is electroplated out of a slightly acidic bath and results in a tin-zinc composition containing from 15-25% zinc. Further details about the process are presented in Appendix B.

2.2.7 Brush Electroplated LHE Cadmium Development

Repair of damaged cadmium coatings on landing gear and other high strength steel surfaces used to require disassembly, cadmium electroplating, and then a baking operation at 190°C (375°F) for between 4-24 hours to alleviate hydrogen embrittlement concerns. When a low hydrogen embrittlement (LHE) cadmium brush plating solution was developed in the late 1960's which did not require a bake after application, thereby eliminating expensive and time consuming disassembly of the component, manufacturing industries took notice. Numerous aircraft and landing gear manufacturers began qualifying and adding the LHE brush cadmium plating process to their specifications. The original LHE cadmium plating process was a registered trademark of Selectrons Ltd., which was acquired by SIFCO Industries Inc. in 1992 [26]. The product selected for testing in this project was SIFCO 2023 LHE Brush Cadmium.

2.2.8 Repair Coating Alternatives Development

Brush electroplated zinc-nickel was developed by SIFCO Applied Surface Concepts as an alternative to brush cadmium around 2005 due to DoD depot and OEM interest. The brush electroplated tin-zinc coating had been developed by Liquid Development Corporation (LDC) for applications other than steel substrates, but was not considered as a cadmium alternative for high-strength steels until the JCAT chose alternatives for this project. The Sermetel[®] 249/273 Al-ceramic product had been developed by Sermetech as a sacrificial coating touch-up which does not need to be heat cured. Sermetech manufactures other high temperature resistant coatings which typically need to be oven cured, although these would not be suitable for field level repair and general touch-up operations. The application of SermeTel[®] 273 after SermeTel[®] 249 effectively cures the coating which makes it viable for field level coating touch-up/repair. Process details for each of these three alternative repair methods are more completely listed in Appendix B.

2.3 ADVANTAGES AND LIMITATIONS OF THE TECHNOLOGY

Process and ESOH advantages and disadvantages for each cadmium alternative coating system as compared to the baseline processes are provided in Table 3. Each alternative offers the potential benefit of eliminating cadmium in select applications. Essentially the electroplating based alternatives (LHE Zn-Ni and AlumiPlate™ coating) can potentially offer similar component coverage as plated cadmium when using auxiliary anodes to coating internal recessed surfaces while the sputtered aluminum was specifically designed for larger IDs of high strength steel components.

Table 3. Advantages and Disadvantages of Each Alternative

Coating	Coating Process Specification or Product	ESOH and Process Advantages	Coating Process or ESOH Disadvantages
LHE Cadmium with Cr(VI) post-treatment	MIL-STD-870, AMS 2401	Baseline HazMat – Coating process is very mature and known	Cadmium plating; cadmium, cyanide; Post-treatment: Cr(VI)
IVD Aluminum	MIL-DTL-83488 Class 2, AMS 2427	Eliminates cadmium for some applications; Many DoD depots already own/operate this equipment.	Less complete coverage than plating; May still require Cr(VI) post-treatment
Magnetron Sputtered Aluminum	Marshall Labs Process; MIL-DTL-83488 Class 2	Eliminate cadmium for some applications; Technology was already being demonstrated in parallel Air Force project	Generally less complete coverage than plating; May still require Cr(VI) post-treatment
Electroplated Aluminum (AlumiPlate™)	AlumiPlate™ process specification; MIL-DTL-83488 Class 2	Eliminate cadmium for some applications; maintain ID coating coverage where necessary; field tests a limited	Process is more capital intensive and involved some ESOH risks; May still require Cr(VI) post-treatment
Zinc-Nickel (Dipsol LHE)	Boeing/Dipsol Procedure for IZ-C17 plating; AMS 2417	Eliminate cadmium for some applications; Maintain ID coating coverage where necessary	Waste treatment for Zn and Ni species required; May still require Cr(VI) post-treatment
Repair Coating Alternatives			
Brush Cadmium	SIFCO 2023	N/A - Baseline HazMat	Baseline HazMat; Uses Cr(VI) post-treatment
Brush Zinc-Nickel	SIFCO 4018	Eliminate cadmium	May still require Cr(VI) post-treatment
Brush Tin-Zinc	LDC 5030	Eliminate cadmium	May still require Cr(VI) post-treatment
SermeTel® 249/273	Sermetech Engineering Bulletin 249	Eliminate cadmium; Does not require capital (rectifier) to apply; less operator skill required	Local coating application with least operator skill level

3.0 PERFORMANCE OBJECTIVES

Due to the original scope of WP-0022, performance objectives for the cadmium alternative coatings evaluated in this project were largely based on comparing each candidate coating's performance in the extensive laboratory testing conducted to both cadmium and IVD-Al control coatings' performance. There was no requirement for field demonstrations in this project as it was structured to rigorously test these alternatives according to the developed cadmium alternative JTP; during or even prior to the course of this test project OEMs and military depots had initiated various demonstrations and implementations of some of these technologies so in essence some of these coatings/processes had already met a certain level of cost and performance expectation (Sections 4 and 8 will include brief descriptions of these other efforts). With respect to the JTP test results, some ratings were made with simple Pass/Fail criteria although more quantitative relative assessments for each performance parameter were provided wherever possible to better discern relative coating performance differences. Table 4 represents a top level summary of performance objectives for making implementation decisions. Qualitative performance benefits listed in the table are an important part of implementation decisions along with the quantitative technical comparisons presented here, and these will be discussed in a relatively general manner since no one specific coating alternative was demonstrated in this project.

Along with the ESOH benefits of the cadmium alternative coatings being compared in this study there are process advantages and/or disadvantages for each. Essentially the electroplating based alternatives, Low Hydrogen Embrittlement (LHE) Zn-Ni (IZ-C17 process) and AlumiPlate™ coatings could offer similar component coverage as plated cadmium when using auxiliary anodes to coat internal recessed surfaces, while the sputtered aluminum process was specifically designed to provide sacrificial coating for larger internal diameters of U.S.A.F. landing gear due to the known limitation of Ion Vapor Deposited Aluminum (IVD-Al) to coating into recessed areas. No single alternative investigated here is yet authorized to replace cadmium in the varied cross-DoD applications on high strength steel.

Table 4. Performance Objectives for the Alternative Coating Systems

Performance Objective	Data Requirements	Success Criteria	Results
Quantitative Performance Objectives			
<u>Coating Performance</u> <ul style="list-style-type: none"> - Embrittlement Resistance - Bend Adhesion - Corrosion Resistance - SCC Resistance - Paint Adhesion - Fatigue testing (Air and salt water) - Torque-tension - Run-on / Breakaway torque - Re-workable - Repairable 	<ul style="list-style-type: none"> • Full range of coatings tests specified in the JTP, as executed here 	Individual JTP performance test criteria are to be evaluated per JTP common and extended performance criteria; Weighting factors for individual test results may be applied differently for each design authority's Implementation Decisions. Formal weighting factors are beyond scope of this project	See Sec. 6.1 for a thorough evaluation of JTP performance parameter results; no single coating performed \geq Cd in all JTP tests thus interpretation is not straight forward
<u>Process Benefits</u> <ul style="list-style-type: none"> - Enhanced coverage - ESOH Improvements - Cost reduction 	<ul style="list-style-type: none"> • Throwing Power • Compliance with OSHA PELs, Other U.S. Regs, International Regs • Coating cost per unit area 	Throwing power test as described in JTP; Coating cost must not be substantially greater than cadmium (ideally less) although life-cycle costs should be primary cost driver	LHE Zn-Ni, Sputtered Al, and AlumiPlate™ all offer certain enhancements over IVD-Al control coatings;
Reduction of hazardous process steps and hazardous waste generated	<ul style="list-style-type: none"> • Cost of waste 	Elimination of cadmium; potential to eliminate Cr(VI) post-treatment	Vacuum based Al coatings are most environmentally compliant; LHE Zn-Ni is better than LHE Cd (cyanide)
Qualitative Performance Objectives			
Drop-in Replacement for Cadmium Processing Infrastructure	<ul style="list-style-type: none"> • Compare alternative process to LHE Cd plating 	Process facility requirements similar to that required for LHE cadmium	Only LHE Zn-Ni meets this criterion
Compatible with IVD-Al Processing Infrastructure	<ul style="list-style-type: none"> • Compare alternative vacuum applied coating process to IVD-Al 	Coating provides supplemental coverage over IVD-Al with acceptable coating performance per JTP	Magnetron sputtered Al provides this capability, but was not equivalent in performance to IVD-Al in JTP tests (Phase II H-RE, although it performed >IVD-Al in Army 'C-ring' H-RE /SCC testing)

4.0 SITE/PLATFORM DESCRIPTION

4.1 TEST PLATFORMS/FACILITIES

There has been strong OEM and military depot interest for each of the alternatives tested in this project due to the unique characteristics of each and it is acknowledged that various levels of demonstration activities were running concurrently with this Joint Test effort. While some of these were neither directed nor funded fully through this effort details will be provided where permitted and as applicable.

4.1.1 LHE Zn-Ni (IZ-C17 process) Plating Demonstrations

There was a plan to conduct a leveraged demonstration of the LHE Zn-Ni coating (IZ-C17 process) being tested in this project at the Navy's FRC-SE, however, it was placed on hold pending resolution of concerns about the coating readiness for Navy demonstration, specifically completion of the Phase II test results. Plating tank modifications had been initiated in 2008 using leveraged funding from JG-PP (Fig. 10) but the effort was placed on hold while the U.S. Air Force took the lead on generating the additional data needed for successful scale-up. The Navy demonstration is being re-engaged in the current fiscal year (FY11), however, it will provide further necessary comparisons of both Cr(VI) and non-Cr(VI) post-treatments while building on the technical data already generated herein. Test emphasis will be on the more highly weighted Navy preferred tests and protocols.

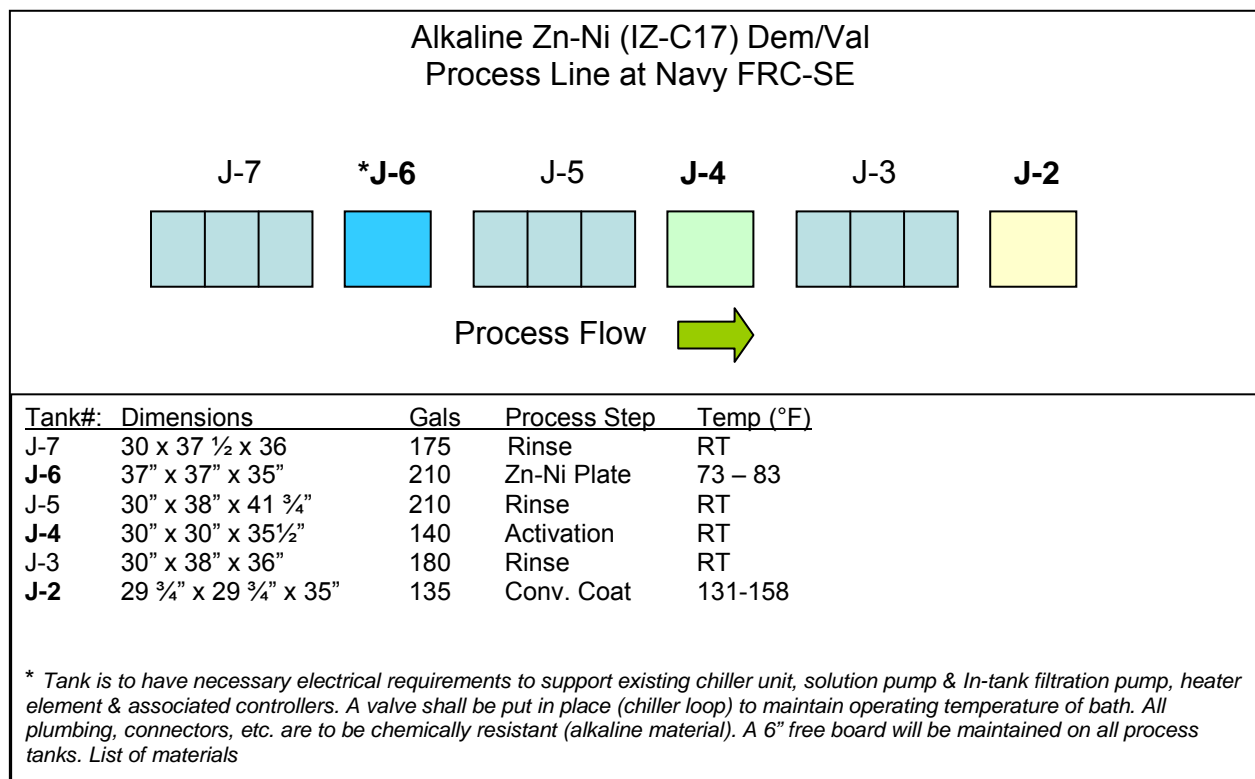


Figure 10. Initial Concept for LHE Zn-Ni Process line at Navy FRC-SE

Additional tests will assess performance in SCC and corrosion fatigue in the unpainted condition and also with chromate and non-chromate primers, and will also include Cr(VI) and Cr(III) post-treatments. The Air Force led demonstration of LHE Zn-Ni also included non-Cr(VI) post-treatments. One significant advantage of the trivalent chromium post-treatment is that due to its inherent thermal stability it can be applied before the baking operation, eliminating process steps.

The Navy's FRC-SE has historically refurbished P-3 and F/A-18 aircraft.

4.1.2 Magnetron Sputtered Aluminum Coating Demonstration

Magnetron sputtered aluminum coatings had been implemented at HAFB/OO-ALC during or prior to the course of this project. As discussed earlier the sputter capability is intended to be augmented to existing IVD-Al vacuum chambers making it a n incremental improvement requiring less capital than stand-alone sputter coating equipment. Limited applications are currently authorized so the equipment experiences intermittent operation.

4.1.3 AlumiPlate™ Coating Demonstration

NAVAIR issued an authorization letter for the AlumiPlate™ coating as a result of the testing performed in this project. The AlumiPlate™ coating was being commercially demonstrated on various military components by other entities prior to the conclusion of this project. For high-strength steels, one of the F-35 variants is being used to field test the AlumiPlate™ coating side-by-side with LHE Cd on selected landing gear components. Other high strength pins and components are also to be included in the demonstration.

The following is a list of reported field testing or implementation of AlumiPlate™ on high-strength steel substrates relevant to this project.

- CH-53K Super Stallion – HSS landing gear
- F-35 – Field test adjacent to Cd on HSS landing gear
- AH-1 Super Cobra – M50 HSS rotor hub housing
- C-5 Galaxy – Aircraft wheel fuse plugs
- DDG-1000 – HSS structural applications, deck hatch hinges
- Howitzer – HSS eye bolts field tested in Hawaii

4.2 PRESENT OPERATIONS

Electroplating of cadmium is typically conducted according to the processing flow illustrated in Fig. 11, while a representative industrial installation for aqueous plating had been shown previously in Figure 3 (Section 2.1). As with any electroplating, the components to be coated are cleaned as appropriate, masked, electroplated and rinsed. Primary sources of hazardous materials include occupational hazards of preparing the bath and adding make-up chemicals, and handling/treating of the contaminated rinse waters. LHE cadmium plating for high-strength steel components (up to 240 ksi) is primarily covered by MIL-STD-870 (Inactive for New Design).

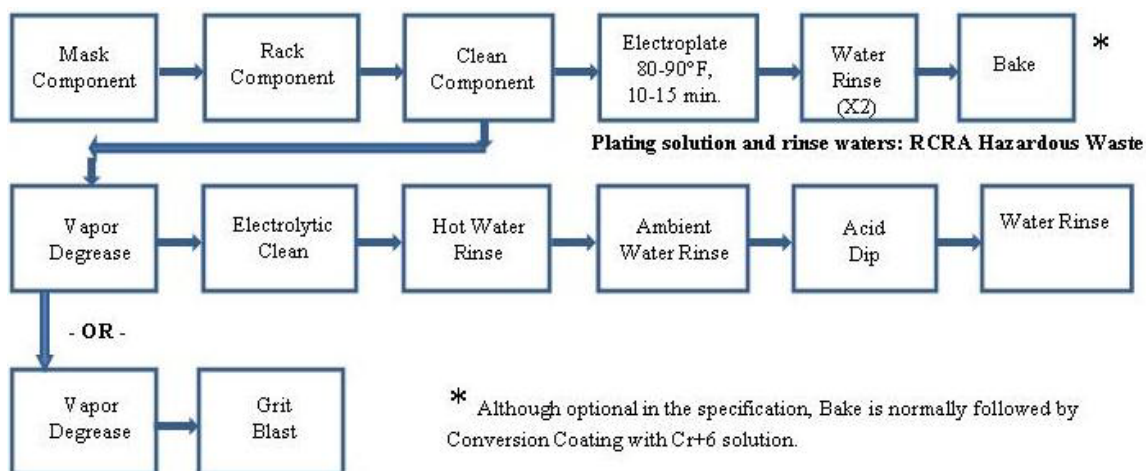


Figure 11. Process Schematic for Existing Cadmium Cyanide Electroplating

4.3 SITE-RELATED PERMITS AND REGULATIONS

There are no permit issues with using either the IVD-Al or the sputtered aluminum coating processes at the DoD facilities presently using these coatings. Cadmium plating operations require worker exposure levels to be determined, and if high enough, a medical surveillance plan implemented. Disposal of cadmium solutions and cadmium waste must be reported to various agencies.

LHE Zn-Ni (IZ-C17 Process) – Implementation of this plating technology would eliminate cadmium and cyanide concerns with respect to both process safety and waste treatment arenas. While there are some waste treatment concerns with both zinc and nickel species in the plating rinse waters and with the spent plating solution itself, these have been addressed to some extent through parallel U.S. Air Force SBIR efforts and not significantly different than the treatments required for industrial Zn-Ni plating solutions.

Electroplated Aluminum – The AlumiPlate™ coating process and facility permitting requirements are the most significant of any alternative considered here due to the hazards inherent in the processing in its current state. Military depots have already indicated that it would be unlikely that permitting would be sought to establish a facility on U. S. government property. Special handling is required for the solutions in the processing line, including the plating solution, make-up chemistry, pre-dips and rinse tanks. Provision must be made for reducing or eliminating volatile organic air pollutants which could be released when the parts are removed from the processing line.

5.0 TEST DESIGN

5.1 JOINT TEST PROTOCOL (JTP) TEST DESCRIPTIONS

This section will describe all of the testing methodologies as performed under the JTP as originally published by the participating stakeholders at that time (31 July 2003) with minor editorial revisions and corrections. Note that original JTP section numbers are preceded by 5.1 in this report (i.e. JTP Sec. 3.1.1 → WP-0022 Final Report Sec. 5.1.3.1.1). Execution of all the tests reflected in the JTP was originally envisioned to be performed in three phases, the first two being reported independently prior to the final report. Those tests which were not executed under ESTCP WP-0022 project will be noted with the JTP test results in Section 6.1 Table 5 provides a listing of the common performance and testing requirements as determined by the working group participants at that time. Table 6 lists the extended performance and testing requirements by the U.S. Army, U.S. Air Force and the U.S. Navy. The organizations tasked with executing each performance test are listed in Table 7.

Table 5. Common Performance and Testing Requirements

Engineering Requirement	Test(s)	JTP Section	Acceptance Criteria	References
Appearance	Visual	3.1.1 → 5.1.3.1.1 in this report	Coating continuous, smooth, adherent, uniform in appearance, free from blisters, pits, nodules, burning, contaminants, excessive powder and other apparent defects	MIL-STD-870; AMS-QQ-P-416
Throwing Power/ Composition Uniformity	Tube fixture / X-ray Fluorescence (XRF)	3.1.2	Alloy composition consistently within process specification range; document any throwing power issues (i.e. coating thickness/composition variation in recesses)	JG-PP JTP BD-P-1-1
Strippability	Coater recommendation or MIL-S-5002	3.1.3	Removal within 2 hours, or time acceptable to manufacturer. Embrittlement by the stripping process is undesirable but may be acceptable. Re-applied coating must meet the acceptance criteria of Sec. 3.2.1 and 3.6.1	MIL-STD-871; ASTM B 571; ASTM F 519
Galvanic Potential	Electrochemical Testing: Open Circuit Potential; EIS / Tafel Analysis	3.1.4	Prefer the OCP of the coating to stabilize within ~24h; Electrochemical impedance spectroscopy exhibits a steady state corrosion rate; No evidence of pitting or crevice corrosion on exposed test panel.	
Adhesion to Substrate	Bend Adhesion Test	3.2.1	No separation (flaking, peeling or blistering) of the coating from the basis metal or from any underplating at the rupture edge. Cracking is acceptable in the bend area if the coating cannot be peeled back with a sharp instrument	ASTM B 571
Paint Adhesion	Wet Tape Paint Adhesion	3.2.2	Rating of at least 4B (or 4A depending on test performed) in 1 day wet tape adhesion test	MIL-PRF-85582; ASTM D 3359

Accelerated Corrosion Resistance	Salt Spray, unscribed	3.3.1	Minimum of 3000h before appearance of red rust or comparable to LHE cadmium controls	ASTM B 117; GM9540P; JG-PP JTP BD-P-1-1
	Salt Spray, scribed	3.3.2	Minimum of 1000h before appearance of red rust or comparable to LHE cadmium controls	ASTM B 117; GM9540P; JG-PP JTP BD-P-1-1
Galvanic Corrosion Resistance	Galvanic couples tested in accelerated corrosion test	3.3.3	Alternative meets or exceeds cadmium in appearance, corrosion resistance and electrical conductivity	ASTM B 117; ASTM G85 Annex 5; JG-PP JTP BD-P-1-1
Fluid Corrosion Resistance	ASTM F 483	3.3.4	No coating degradation greater than that of LHE cadmium controls as determined by weight loss and appearance	ASTM F 483; SAE ARP 1755; SAE AMS 1435
Accelerated Corrosion Resistance	Painted and scribed salt spray resistance	3.3.5	Minimum of 3000h before appearance of red rust or comparable to LHE cadmium controls	ASTM B 117; GM9540P; JG-PP JTP BD-P-1-1
Run-on and Breakaway Torque	NASM1312-31	3.4.1	During installation, maximum locking torque shall not exceed 80 in-lb for -06032 and 300 in-lb for -10032. During removal, the minimum breakaway torque shall not be less than 9.5 in-lb and 32 in-lb, respectively (values from NASM25027); After 15 cycles locking torque test, nut and bolt threads shall remain in serviceable condition: no thread peel, missing segments, cracks, galling, or splits when examined at 10X, these features are unacceptable	NASM1312-31; MIL-STD-870; SAE AMS QQ-P-416; NASM25027
Torque-Tension	NASM1312-15 Torque-Tension method	3.4.2	Torque-tension for candidate material is within the range for cadmium plated threaded parts. Threaded part does not yield or fracture, threads do not strip.	NASM1312-15; GSE Inc. Model 845 torque-tension tester
Torque-Tension for Corrosion Exposed Fasteners	Torque-Tension test including corrosion test exposure	3.4.3	Torque-tension for candidate material is within the range for cadmium plated threaded parts. Removal torque and torque-tension after cyclic corrosion exposure is comparable to unexposed configuration	NASM1312-15; GSE Inc. Model 845 torque-tension tester; GM9540P
Fatigue	R. R. Moore rotating bending fatigue	3.5.1	Fatigue values should be comparable to cadmium plated specimens	ASTM E 468; ISO 1143
Hydrogen Embrittlement	H-E test in Air	3.6.1	NFS of bare and coated specimens within 10ksi of average reported by manufacturer AND 4/4 specimens sustain 75% NFS for 200h sustained load testing (SLT) without fracture – OR—Only 1 of 4 specimens fractures <200h but the remaining 3 sustain at least 1h at 90% NFS	ASTM F 519; ASTM E 8
	Hydrogen Re-Embrittlement test in reagent water, sea water and glycol solution	3.6.2	Average load and time to fracture greater than or equal to cadmium when tested in 1 MO reagent water; criteria for other fluids were not stipulated at time of JTP creation, however, comparison to LHE cadmium is recommended	ASTM F 1624
Repairability	Appearance	3.7.1	Same as for primary coatings, above	JTP Sec. 3.1.1
Repairability	Bend Adhesion	3.7.1	Same as for primary coatings, above	JTP Sec. 3.2.1

Repairability	Thickness	3.7.1	Per applicable plating specification	MIL-STD-870; AMS-QQ-P-416
Repairability	Paint Adhesion	3.7.1	Same as for primary coatings, above	JTP Sec. 3.2.2
Repairability	Corrosion Resistance, unscribed	3.7.1	Same as for primary coatings, above	JTP Sec. 3.3.1
Repairability	Corrosion Resistance, scribed	3.7.1	Same as for primary coatings, above	JTP Sec. 3.3.2

Table 6. Extended Performance and Testing Requirements

Engineering Requirement	Test	JTP Sec.	Acceptance Criteria	References	Participants Requiring Test
Acidified Cyclic SO ₂ Corrosion	Cyclic SO ₂ Salt Spray, unscribed	4.1.1	Equivalent or better than LHE Cadmium	ASTM G 85 Annex 4	NAVAIR
	Cyclic SO ₂ Salt Spray, scribed	4.1.2	Equivalent or better than LHE Cadmium	ASTM G 85 Annex 4	NAVAIR
	Cyclic SO ₂ Salt spray in the scribed, painted condition	4.1.3	Equivalent or better than LHE Cadmium	ASTM G 85 Annex 4	NAVAIR
Corrosion Fatigue	Corrosion Fatigue	4.1.4	Equivalent or better than LHE Cadmium	ASTM E 466	NAVAIR
Lubricity	Run-on / Breakaway Torque with corrosion exposure	4.2.1	Equivalent or better than LHE Cadmium	NASM1312-31; MIL-STD-870; SAE AMS QQ-P-416	U.S.A.F., NAVAIR
SCC Resistance	SCC	4.3	Equivalent or better than LHE Cadmium	ASTM F 1624	NAVAIR
Additional Fatigue	Fatigue specimens of different geometry and/or R-value	4.4	Equivalent or better than LHE Cadmium	ASTM E 466	NAVAIR
U.S. Air Force	Torque-tension	5.1	Comparable with LHE Cadmium	U.S. Air Force	U.S.A.F.
U.S. Army	H-RE / SCC using Type 1d C-rings	6.1	Equivalent or better than LHE Cadmium	AMCOM	AMCOM

Service-specific performance requirements and test methods are defined in this section. Test descriptions, rationales and methodologies are described along with any required major or unique equipment or instrumentation. A data analysis section is also included if the data recorded from the testing is to be manipulated prior to analysis. The test methodology includes the definition of test parameters and conditions, test specimens/substrates, number of trials, and acceptance criteria. The test methods represent acceptable procedures to define a performance requirement or to differentiate performance characteristics between different coatings.

Table 7. Test Facility Identification for Phase I and Phase II JTP Test Efforts

Test Category	Test	Testing Facility
Phase I – Primary Coatings		
General Properties		
Adhesion	Bend Adhesion	NAVAIR
Embrittlement	Hydrogen Embrittlement	NAVAIR
	Hydrogen Re-embrittlement	NAVAIR
	Army AMCOM H-RE/SCC ‘C-ring’ tests	ARL
Phase II – Primary and Repair Coatings		
General Properties (primary coatings)	Appearance	CTC
	Thickness	CTC
	Throwing power and alloy composition uniformity	CTC
	Strippability	NAVAIR
	Galvanic potential	Not tested
Adhesion (primary coatings)	Bend adhesion	NAVAIR
	Paint adhesion	NAVAIR
Corrosion (primary coatings)	Unscribed neutral salt spray (NSS) (bare)	ARL
	Scribed NSS (bare)	ARL
	Galvanic corrosion resistance	ARL
	Fluid corrosion resistance	ARL
	Scribed w/ primer & topcoat	NAVAIR (paint)/ARL (test)
	SO ₂ Salt Fog	NAVAIR (paint/test)
Lubricity (primary coatings)	Run-on/break-away torque	WMTR
	Torque-tension	WMTR
Reparability (for repair coatings only)	Appearance	CTC
	Bend adhesion	ARL
	Thickness	CTC
	Paint adhesion	Not tested
	Unscribed corrosion resistance	ARL
	Scribed corrosion resistance	ARL

The types of substrates that were used for testing, unless otherwise specified, are listed in Table 8 along with their respective test specimen code—these codes may be used throughout this section to define substrate types for each test procedure. Specimens for adhesion tests were nominally 1 inch x 4 inches x 0.04 inches. Specimens for corrosion resistance and paint adhesion tests were nominal 4 inches x 6 inches x 0.04 inches unless otherwise specified. The focus of this JTP is on high-strength structural alloy steels used for various applications. Alloy AISI 4130 was used for adhesion and corrosion tests and for general properties determination. Fatigue and H-E tests used specialized coupons. The specific alloy and configuration is listed in each following test description.

Table 8. Test Specimen Codes and Substrate Specifications

Test Specimen Code	Substrate Specification and Condition
AL1	Aluminum alloy 2024-T3, conforming to ASTM B 209-92a
AL2	Aluminum alloy 7075-T73, conforming to ASTM B 209-92a
AL3	Aluminum alloy 6061-T6, bare, conforming to ASTM B 209 - 92a, standard specification for aluminum and aluminum-alloy sheet and plate
LS1	Alloy Steel 4130, conforming to AMS 6350 steel, sheet, and plate
HS1	Alloy Steel 4340, conforming to AMS 6414P steel, bars, forgings and tubing, 260-280KSI
HS2	Alloy Steel 300M, conforming to AMS 6419 steel, bars, forgings and tubing, 280-300KSI
ST2	Corrosion resistant steel alloy 17-4 PH conforming to AMS 5643, Steel, Corrosion Resistant, Bars, Forgings, Rings, and Extrusions
TI1	Titanium alloy, 6% aluminum, 4% vanadium, conforming to SAE AMS T-9046A titanium and titanium alloy sheet, strip, and plate
BR1	Aluminum/Nickel/Bronze Alloy conforming to AMS 4640, Aluminum Bronze, Bars, Rods, Shapes, Tubes, and Forgings
CB1	Copper Beryllium, ASTM B 194 plate, sheet, strip and rolled bar –OR- SAE AMS 4530F sheet, strip, and plate

Apply all candidate coatings per manufacturer's instructions or specification requirements, if available. Application process including thickness, supplemental coatings, post plate baking, etc., shall be those expected for the coating as used in production. Specimens are not peened prior to plating. Unpeened specimens will highlight the effect of coatings on fatigue. Unless otherwise specified, the Cd-plating on control specimens shall conform to MIL-STD-870B (*Cadmium Plating, Low Embrittlement, Electrodeposition*, issued July 30, 1986) Class 2 Type II. Unless otherwise specified the minimum thickness shall be 0.0003 inches with the maximum thickness being that expected during normal application (nominally 0.0006 inches maximum unless other specified).

5.1.3.1.1 Appearance (HSS JTP 3.1.1)

Test Description

Prepare the specimens and apply the coatings. Evaluate coating by observing color, texture, and uniformity of appearance by unaided visual inspection. Using the coating manufacturer's guidance, acceptable color levels for a particular coating system can be developed utilizing color chips. Measure the coating thickness at several locations. Record and report any coating defects observed. Test methodology is summarized in Table 9.

Rationale

MIL-STD-870B and FED-STD-QQ-P-416F (*Plating, Cadmium (Electrodeposited)*) require Cd-plating to be smooth, adherent, uniform in appearance, and free from blisters, pits, nodules, burning and other defects. These requirements are also applicable to candidate coatings.

Table 9. Test Methodology for Coating Appearance

Parameters	Unaided visual inspection
Type/Number of Specimens	Three alternative plated LS1 specimens
Experimental Control Specimens	None
Acceptance Criteria	Coating must be continuous, smooth, adherent, uniform in appearance, free from blisters, pits, nodules, burning, contaminants, excessive powder, and other apparent defects.
Reference Document	MIL-STD-870B, AMSQQP416

5.1.3.1.2 Throwing Power and Alloy Composition Uniformity (HSS JTP 3.1.2)

The ability of a process to coat complex shapes including the inside of blind holes while maintaining a consistent composition is critical to successful application on complex components. This test uses a fixture to create a cavity on a flat substrate surface. After coating, the substrate can be removed and the thickness and composition of the coating inside the cavity can be determined. Composition of the coating is determined by the energy or wavelength dispersion of the secondary X-ray emissions emanating from the test specimen coating that has been excited to fluorescence with a high-energy X-ray tube. Test methodology is summarized in Table 10.

Test Description

Fixture: Fixtures were constructed to the dimensions shown in Figure 12. The fixtures were made from Teflon, with the exception of the fixtures for electroplated Al. These fixtures were made by the vendor to the same dimensions to ensure that the fixture would not react with the electroplating bath. In both cases, the fixtures were constructed from a section of pipe with one capped end. Slots were cut into the interior to allow a 3 inch x 5 inch test panel to slide snugly into the pipe. A threaded open cap closed off the fixture. Test panels were placed in the fixtures with caps and any electrical connections installed. Coatings were applied by the respective manufacturer, with the fixture aligned at different orientations, where possible.

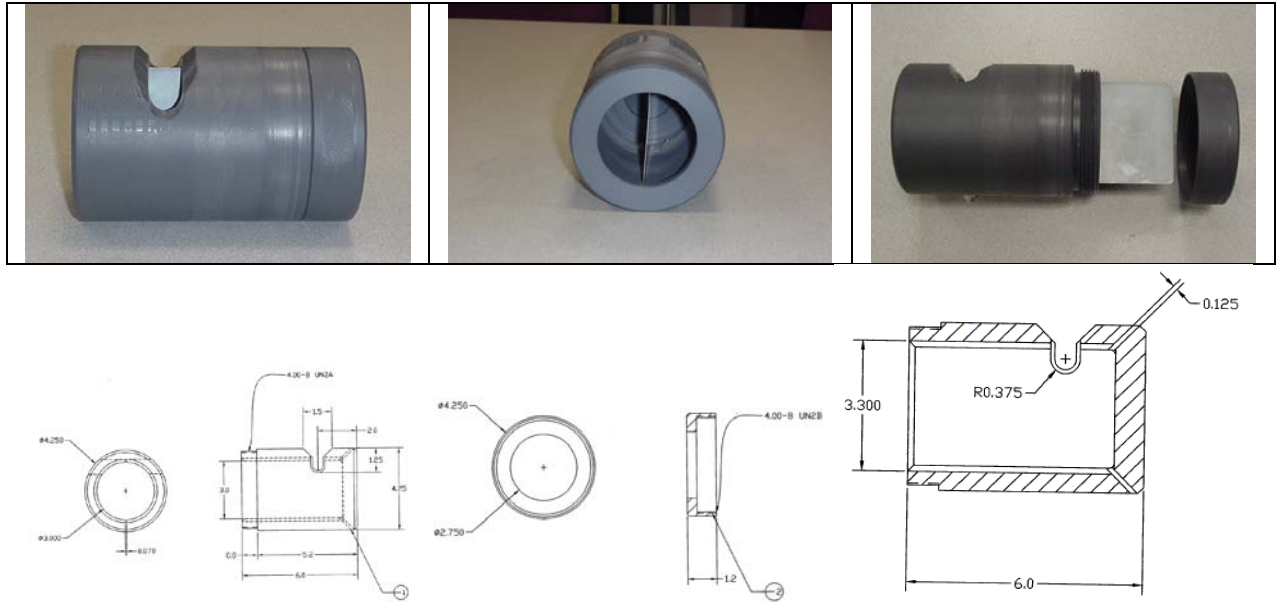


Figure 12. Fixture for “Throwing Power” and Alloy Composition Test

Slide the test panel into the fixture; install cap and any electrical connections. Apply coating IAW the manufacturers recommendations. Place the fixture in the coating chamber so that the desired orientation is achieved and any impediments to coating such as air bubbles, etc., will be minimized. Multiple specimens might be used to fully evaluate a process.

Coating Thickness/”Throwing Power” After coating, remove the test panel from the fixture and determine coating thickness using a micrometer or other measuring device with sufficient precision to accurately measure the coating. Measure thickness at several locations along the panel to determine “throwing power” for the process.

Coating Composition For alloy coatings determine coating composition using X-ray fluorescence. Calibrate and operate the X-ray fluorescence test equipment IAW the manufacturer’s instructions, ASTM E 1621 (*Standard Guide for X-ray Emission Spectrometric Analysis*, dated 1994) and ASTM B 568 (*Standard Test Method for Measurement of Coating Thickness by X-ray Spectrometry*, issued November 15, 1991). Use standards representing various thickness and alloy compositions to calibrate the equipment. Use a beam collimator to restrict the beam size and ensure correct alignment of the beam, specimen, and detector. For energy-dispersive systems, the sample area must be coordinated with the X-ray tube power input to ensure the count-rate capacity of the detector is not exceeded; exceeding the detector’s count-rate capacity may cause dead-time losses. It is recommended to use a sufficient measuring time to collect at least 10,000 counts for every constituent element, in the interest of analytical precision.

For coating consistency, measure composition at least three locations on each test specimen surface (front or back, separated by at least a one inch distance) to determine consistency across the surface. The coating should maintain a consistent composition over the surface of the test specimen. Measure the coating at several points along the fixture cavity to determine

compositional changes that occur as the current density changes along the cavity. The coating should maintain a consistent composition along the fixture cavity.

Rationale

The composition and thickness of coatings is critical to their performance. Consistent composition is required of all coatings. Understanding how far coatings will “throw” into holes and cavities will determine the applicability of a process to potential hardware.

Table 10. Test Methodology for Throwing Power / Composition Testing

Parameters	Measured on at least three (3) locations on each test specimen surface, separated by 1 inch. The total measured area shall be greater than 1 square inch.
Type/Number of Specimens	Three specimens, 4130 steel with fixtures at different orientations to the coating “chamber” geometry.
Experimental Control Specimens	LHE-Cd (MIL-STD-870B or equivalent). Three (3) LS1 with fixtures at different orientations to the coating “chamber” geometry.
Acceptance Criteria	Composition stays within the process specification requirements. Document thickness variation as a function of depth into fixture and orientation in coating process
Reference Document	MIL-STD-870B, AMSQQP416

Major or Unique Equipment

Fixture to create cavity on flat specimen.

X-ray fluorescence test equipment (X-ray tube, beam collimator, detector)

Data Analysis

Convert the resulting energy/wavelength spectrum of the detected secondary emissions to units of composition. Report pertinent coating process details such as the orientation of the fixture relative to the source of the coating material.

5.1.3.1.3 Strippability of Coatings (HSS JTP 3.1.3)

This test evaluates the ability to remove the candidate coating and replace it as may be necessary during the manufacture or repair of a part. This test is necessary to ensure that candidate coating can be removed, replaced, and still meet the requirements for acceptable adhesion to the substrate and that stripping and re-coating will not cause embrittlement of the substrate. The embrittling characteristic of the stripping process is also evaluated. Test methodology is summarized in Table 11.

Test Description

Apply coatings to test specimens as recommended by the respective manufacturers. Remove coatings from specimens as recommended by the manufacturer or IAW MIL-S-5002D (*Surface Treatments and Inorganic Coatings for Metal Surfaces of Weapons Systems*, dated March 24, 1994) in a manner that does not damage the substrate material. Note the amount of time required

to remove 100% of the coating. Verify complete removal of the coating using visual observation, a spot test, or appropriate analytical methods that do not compromise the coupons for further testing or use a witness panel.

Test four of the eight stripped notch tensile specimens for embrittlement IAW Section 5.1.3.6.1. Do not bake prior to testing. Begin embrittlement testing within six (6) hours of stripping specimens. Document any embrittlement noted on the stripped coupons.

The other four tensile stripped tensile specimens and the bend specimens are to be recoated with each alternative coating, including any required baking or post processing operations per the coating supplier. Surfaces shall meet requirements of MIL-S-5002D (*Surface Treatments and Inorganic Coatings for Metal Surfaces of Weapons Systems*, dated March 24, 1994) prior to refinishing.

Test the three re-coated bend specimens IAW Section 5.1.3.2.1 and the four re-coated tensile specimens IAW Section 5.1.3.6.1.

Table 11. Test Methodology for Strippability Testing

Parameters	Specimens stripped of candidate coating, recoated, and tested for adhesion per Section 5.1.3.2.1 - Bend Adhesion and embrittlement per Section 5.1.3.6.1 – Hydrogen Embrittlement
Type/Number of Specimens	Three 4130 steel bend specimens (1 inch x4 inches) and eight H-E bars
Experimental Control Specimens	None
Acceptance Criteria	Candidate coating should be removed in two (2) hours or less using appropriate removal method. Substrate surface after coating removal must meet requirements of MIL-S-5002D prior to refinishing. Embrittlement by the stripping process is undesirable but may be acceptable. Re-applied coating must meet the acceptance criteria of Section 3.2.1 - Bend Adhesion and 3.6.1 HE
Reference Document	MIL-STD-870B, AMSQP416

5.1.3.1.4 Electrochemical Galvanic Potential (HSS JTP 3.1.4)

This test will determine a galvanic potential value that will allow comparison of these alternative coatings to other materials in the electrochemical galvanic series.

Test Description

Four (4) tests are to be conducted:

- Open circuit potential (OCP) to determine steady state conditions
- Electrochemical impedance spectroscopy (EIS) along with equivalent circuit analysis (ECA) to model any corrosion processes present

- Tafel analysis to provide kinetic data for corrosion processes
- Visual examination to confirm electrochemical methodologies

Test Panel Preparation -- The 4 inches x 6 inches sheet substrate shall be a 4130 steel or equivalent for all inorganic coating. The candidate inorganic coatings shall be applied on the substrate IAW appropriate specifications or manufacturing protocol.

Open Circuit Potential Measurement -- The test panel shall be inserted into a Princeton Applied Research (PAR) model K0235 Flat Cell or equivalent cell, compatible with performing standard electrochemical testing. The exposed surface area shall be approximately 16 cm². For each panel, the cell shall be filled with fresh, air-equilibrated 5% NaCl solution. For reference, the OCP of the panel shall be measured versus a saturated calomel electrode (SCE). The OCP shall be recorded continuously for 5 days or longer to obtain a steady state OCP, but no longer than 7 days. Ideally, the steady state OCP of each coating shall be positioned into a galvanic series format.

Electrochemical Impedance Spectroscopy -- On days 1, 3 and at end of OCP measurement, an EIS spectrum shall be generated. OCP monitoring will need to be momentarily interrupted for the EIS testing. The applied alternating voltage shall be 15 mV, which should have no significant effect on OCP monitoring. A typical EIS system will include a potentiostat-galvanostat, an impedance/gain-phase analyzer, and a personal computer. The frequency range will be from 1.6E-2 to 1.0E+5 Hz. EIS data shall be taken typically at five (5) selected frequencies per decade. The EIS spectrum shall be interpreted with any equivalent circuit analyzer (ECA) software currently available. The intention of the ECA is to calculate the corrosion rate and to show that the corrosion process is at steady state.

Tafel Analysis -- The cathodic and anodic potentiodynamic scans shall be performed after the completion of OCP measurement and final EIS spectrum generation. The exposed panel shall be cathodically polarized from the OCP to typically - 400 mV versus the OCP (but not to exceed a current density of 1E-4 A/cm²) and returned to the OCP. Then, the OCP of the panel shall be recorded versus time. When the OCP of the panel is at steady state again (less than 1 mV change per 30 minutes), the panel shall be anodically polarized from the OCP to typically +100 mV versus the OCP and returned to the OCP. A typical scan rate for anodic and cathodic polarization is 0.05 mV/sec. The corrosion rate shall be determined from the scans using Tafel extrapolation.

Visual Examination -- Panels shall be removed from the cell at the end of the OCP monitoring, rinsed in deionized water, and gently air-dried. The panel shall be visually and microscopically examined for pits, crevice corrosion (particularly around the perimeter), and discoloration. The panel shall be scanned and stored for reporting, if required.

Rationale

This test is necessary to allow comparison of electrochemical properties of candidate coatings to the properties of LHE-Cd. Test methodology is summarized in Table 12.

Table 12. Test Methodology for Electrochemical Galvanic Potential Testing

Parameters	OCP Measurement	EIS / Tafel Analysis	Visual Examination
Number and Type of Specimens per Candidate	Minimum 2 LS1 panels tested per coating candidate	Minimum 2 LS1 panels tested per coating candidate	Minimum 2 LS1 panels tested per coating candidate
Experimental Control Specimens	Minimum 2 LS1 panels with LHE-Cd	Minimum 2 LS1 panels with LHE-Cd	Minimum 2 LS1 panels with LHE-Cd
Acceptance Criteria	Prefer the OCP of the coating to achieve steady state in less than 1 day.	Steady state corrosion rate.	No evidence of pitting or crevice corrosion on exposed test panel

Major or unique equipment

Personal computer with electrochemical and ECA software

Potentiostat-galvanostat

Impedance/gain-phase analyzer

Electrochemical test cell

Data Analysis

The OCP for each material shall be plotted versus time. In a typical plot, the OCP will become independent of time or, in other words, the OCP will reach steady state. The attainment of a steady state OCP may take up to 24 hours or more. In some cases, the OCP may appear to wander with time; the test coupon should be examined for corrosion around the edge and for pitting. In either case, the test should be rerun. The steady state OCP of the materials shall be used for generation of the galvanic series. Report corrosion rate derived from Tafel curves.

5.1.3.2.1 Bend Adhesion (HSS JTP 3.2.1)

Test Description

This test evaluates the adhesion of a coating to several different common alloy substrates. Table 13 summarizes the test methodology for this test.

Table 13. Test Methodology for Bend Adhesion Testing

Parameters	Specimen is bent back and forth through 180° until the coating and/or substrate ruptures
Type/Number of Specimens	3 specimens of each of the following substrate materials: 4130 steel, 17-4 PH stainless steel, 6Al-4V Titanium
Experimental Control Specimens	None required
Acceptance Criteria	No separation (flaking, peeling, or blistering) of the coating from the basis metal or from any under-plating at the rupture edge. Cracking is acceptable in the bend area if the coating cannot be peeled back with a sharp instrument.
Reference Document	MIL-STD-870B, AMSQQP416

Apply alternate coatings to test specimens as recommended by the manufacturers. Clamp a specimen in a vise and then bend the projecting end back and forth until rupture of the basis metal and/or coating occurs, IAW ASTM B 571-91 (*Standard Test Methods for Adhesion of*

Metallic Coatings, issued February 22, 1991). Examine the edge of the ruptured coating under four times magnification for peeling or flaking of the coating from the substrate.

Rationale

The acceptance criteria for this adhesion test match the requirements specified in MIL-STD-870B and AMS QQ-P-416 (*Plating, Cadmium, (Electrodeposited)*, issued July 2000). This test is necessary to qualify candidate coatings for acceptable adhesion to the substrate.

5.1.3.2.2 Wet Tape Paint Adhesion (HSS JTP 3.2.2)

This test assesses the general adequacy of paint adhesion to flat surfaces coated with the candidate coating. The test is conducted by applying and removing pressure-sensitive adhesive tape over scratches made through the paint. Test methodology is summarized in Table 14.

Test Description

Perform this wet tape adhesion test IAW ASTM D 3359 Method B (*Standard Test Methods for Measuring Adhesion by Tape Test*). Apply coating to test specimens as recommended by the manufacturer. To one (1) set of specimens, apply a waterborne epoxy primer, conforming to MIL-PRF-85582 Type I, Class C1 (*Primer Coatings: Epoxy, Waterborne*), and allow to dry in air for 14 days prior to testing. To another set of specimens, apply a non-chromated waterborne epoxy primer, reference MIL-PRF-85582 Type I Class N (e.g., PRC Desoto/Spraylat EWAE118 A/B Type II, Class N or Akzo Nobel 10PW22-2/ECW119) and allow the primer to dry in air for 14 days prior to testing. To a third set of specimens, apply a solvent borne primer conforming to MIL-PRF-23377 Type I, Class C (*Primer Coatings: Epoxy, High Solids*) and allow to dry in air for 14 days prior to testing. Other primers may be used as determined at the time of testing.

For each primer type, immerse sets of 3 candidate and three control specimens in distilled water at each of three conditions: 24 hours at 23°C (73°F), 96 hours at 49°C (120°F), and for 168 hours at 65°C (150°F). At the conclusion of exposure, remove the specimens and wipe dry with a soft cloth. Within one (1) minute of removal from the water, scribe through the coating to the basis metal in a grid pattern IAW ASTM D 3359 Method B. Immediately, apply a 1 inch wide strip of masking tape (average adhesion of 60 ounces per inches width) centered on the grid. Press the tape with a pencil eraser, or other appropriate tool, until the tape is firmly adhered to the coating. Remove the tape with one quick motion and examine for coating adhesion.

Rationale

This test conducted at 23°C (73°F) is necessary to qualify candidate coatings for use on substrates that may be painted. Elevated temperature testing conducted at 49°C (120°F) and 65°C (150°F) did not have acceptance criteria established as common performance requirements, however, military primer specifications typically include elevated temperature water immersion tests for the primer on conversion coated aluminum surfaces (e.g, MIL-PRF-85582 (4 days at 120°F); TT-P-2756 (7 days at 150°F).

Table 14. Test Methodology for Wet Tape Paint Adhesion Testing

Parameters	Immerse separate specimens in distilled water for 24 hours and 168 hours at 23°C (73°F), 96 hours at
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	49°C (120°F), and 168 hours at 65°C (150°F), respectively.
Type/Number of Specimens	Three 4130 specimens per primer per time/temperature combination
Experimental Control Specimens	Three Cd plated 4130 specimens per primer per time/temperature condition
Acceptance Criteria	Adhesion not less than 4B as determined using the criteria in ASTM D 3359 for specimens immersed for 24 hours at 23°C.
Reference Document	MIL-STD-870B, AMSQQP416

5.1.3.3.1 Unscribed Salt Spray (Fog) Corrosion Resistance (HSS JTP 3.3.1)

This test evaluates the ability of a candidate coating to prevent corrosion of coated substrates exposed to neutral salt spray conditions conducted IAW ASTM B 117. Test methodology is summarized in Table 15.

Test Description

Apply coating to test specimens. Place test specimens in a salt spray chamber operated IAW ASTM B 117-94 (*Standard Practice for Operating Salt Spray (Fog) Testing Apparatus*, approved February 15, 1994). Examine panels in the test chamber weekly and record appearance of corrosion products. Monitor the first appearance and progress of white and black corrosion products. Rate the panels IAW ASTM D 1654. Remove the specimens from the salt spray chamber when examination reveals red rust.

Rationale

This test is necessary to qualify candidate coatings for use on substrates not resistant to corrosion.

Table 15. Test Methodology for Unscribed Salt Spray Testing

Parameters	Conduct salt spray test IAW ASTM B 117: 5% sodium chloride (NaCl) solution sprayed at chamber temperature of 35°C (95°F) until coating failure. Angle panels at 6° off normal.
Type/Number of Specimens	Three 4130 specimens per candidate coating
Experimental Control Specimens	Three LHE Cd plated 4130 specimens
Acceptance Criteria	Minimum of 3,000 hours exposure before appearance of red rust or comparable to LHE Cd. Record observations of first appearance and progression of white and black corrosion products.
Reference Document	MIL-STD-870B, AMSQQP416

Major or Unique Equipment

Salt spray chamber operated per ASTM B 117.

5.1.3.3.2 Scribed Salt Spray (Fog) Corrosion Resistance (HSS JTP 3.3.2)

This test evaluates the ability of a candidate coating to prevent corrosion of coated and scribed substrates exposed to neutral salt spray conditions. Test methodology is summarized in Table 16.

Test Description

Apply coating to test specimens. Machine each specimen from corner-to-corner forming an “X” pattern with a 0.030 - 0.060 inches wheel cutter with a “V” cut down to a depth of plating thickness plus 0.010 ± 0.001 inch. Place test specimens in a salt spray chamber operated IAW ASTM B 117-94 (*Standard Practice for Operating Salt Spray (Fog) Testing Apparatus*, approved February 15, 1994). Examine panels in the test chamber weekly and record appearance of corrosion products. Monitor the first appearance and progress of white and black corrosion products. Rate panels IAW ASTM D 1654. Remove the specimens from the salt spray chamber when examination reveals red rust.

Rationale

This test is necessary to qualify candidate coatings for use on substrates not resistant to corrosion.

Table 16. Test Methodology for Scribed Salt Spray Testing

Parameters	Conduct Salt Spray test IAW ASTM B 117: 5% NaCl solution sprayed at 35°C until coating failure. Angle panels at 6° off normal.
Type/Number of Specimens	Three 4130 specimens
Experimental Control Specimens	Three 4130 specimens
Acceptance Criteria	Minimum of 1,000 hours exposure before appearance of red rust or comparable to LHE Cd. Record observations of first appearance and progression of white and black corrosion products.
Reference Document	MIL-STD-870B, AMSQQP416

Major or Unique Equipment

Salt spray chamber, ASTM B 117

5.1.3.3.3 Galvanic Corrosion Resistance (HSS JTP 3.3.3)

Providing galvanic corrosion protection and compatibility between the dissimilar metals used on high-strength steel components and assemblies, such as landing gear, is a critical function of candidate coatings. This test is necessary to ensure candidate coatings provide adequate galvanic corrosion protection to dissimilar metal systems containing two common aerospace aluminum substrates.

Test Description

This test requires that galvanic corrosion test assemblies be fabricated as shown in Fig. 13 using the parts list provided in Table 17. Two of the actual test assemblies for this effort are shown at the bottom of Fig. 13. Apply LHE Cd control and candidate coatings as recommended by the

F. Anodized Washer

E. Nut

D. Washer plated with cadmium or candidate coating

C. Test Block

B. Epoxy Coating

F. Anodized Washer

A. Fastener

Nylon Insert

Table 17. Recommended Parts List for Galvanic Assembly

A	Fastener:	¼-28 x 7/8 Hex Cap Screw or equivalent, stainless steel
B	Epoxy Primer:	MIL-PRF-85582 Type I, Class N
C	Test Block:	0.125-0.250 in. thick AL1 and AL2
D	Test Washer:	Fabricate test washers from 1/8" sheet stock. Washers are to be the same size, ~1" diameter with ~0.266" hole centered on washer sized to accommodate nylon insert. Fabricate washers from LS1, ST2, CB1, and BR1 materials.
E	Nut:	1/4-28 Lock Nut or equivalent, stainless steel
F	Anodized Washer:	Anodized aluminum (NAS1149D0463K) to back nut (E)
F	Anodized Washer:	Anodized aluminum (NAS1149D0463K)
G	Nylon Insert:	¼ Nylon Insert. Length equal to test block + washer thickness

41

Abrade block surface and clean the interface between **C** and **D** with isopropyl alcohol (IPA) just prior to installation.

Fabricate the test assembly as shown in Fig. 13. A nylon insert, **G**, isolates the test block and washer from the fastener. Tighten the nut, **E**, so that the arrangement is firmly held together (a torque reading of 70-80 in-lb for a 1/4-28 size nut). Make a wide scribe through the coating to the substrate on top of washer, **D**. Place one ohmmeter probe in the scribe on washer **D** and place the other probe on the test block, **C**, as shown in Figure 14.

A reading of the electrical resistance should be one milliohm or less for conductive coatings (tighten nut, **E**, and re-clean if it is not). Record the resistance.

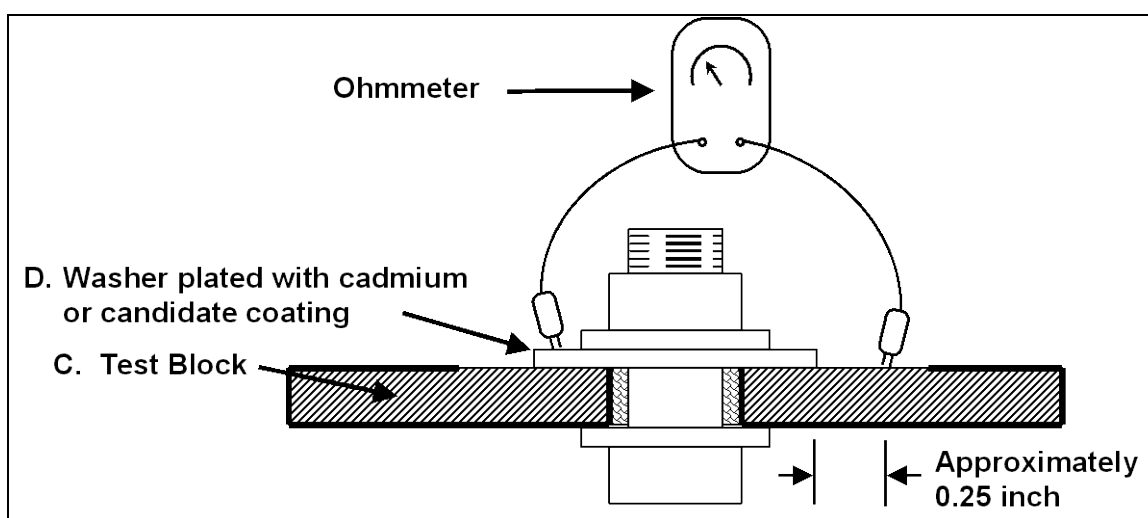


Figure 14. Galvanic Corrosion Resistance Measurement

Exposure and Measurement

Place the test assemblies in a salt spray chamber and perform a spray test IAW ASTM B 117-94 (*Standard Practice for Operating Salt Spray (Fog) Testing Apparatus*, approved February 15, 1994) for 168 hours. Place duplicate assemblies in a cyclic corrosion chamber and perform corrosion testing IAW ASTM G 85 Annex 5 (*Standard Practice for Modified Salt Spray (Fog) Testing: Dilute Electrolyte Cyclic Fog/Dry Test*) for 336 hours.

Remove test assemblies from the corrosion chamber and rinse to remove the excess salt. Allow rinsed test assemblies to dry 3-5 hours in air.

Place one ohmmeter probe in the scribe on the washer, **D**, and place the other probe on the test block, **C**, as shown in Figure 14. If necessary to assure good probe connections, use sandpaper to remove corrosion from the test block, **C**, and washer, **D**. Measure the electrical resistance between the test block, **C**, and the washer, **D**. Record the resistance. Note any corrosion products around the washer, **D**. Disassemble; note condition between the block and the washer. Note difference between Cd specimens and candidate specimens.

Table 18. Test Methodology for Galvanic Corrosion Resistance

Parameters	Salt Spray one set of test assemblies for 168 hours (ASTM B 117) and a duplicate set of assemblies in cyclic corrosion for 336 hours (ASTM G 85 Annex 5). Angle panels at 6° off normal.
Type/Number of Specimens	Three (3) test assemblies for each candidate coated washer substrate to be installed in each test block material. (48 total; four (4) washer substrates, three (3) washers for each test block, two (2) test block substrates, two (2) exposure conditions) Note: All candidate and control assemblies may be assembled onto one large test block.
Experimental Control Specimens	Three (3) test assemblies with Cd coated washers of each substrate. Three (3) test assemblies with bare washers of each substrate.
Acceptance Criteria	Alternative meets or exceeds Cd in appearance, corrosion resistance, and electrical conductivity
Reference Document	MIL-STD-870B, AMSQQP416

Major or Unique Equipment

Salt spray chamber, ASTM B 117

Salt spray chamber, ASTM G 85 Annex 5

Milliohm bonding meter, HP4328A or equivalent, with four-point probe

5.1.3.3.4 Fluid Corrosion Resistance (HSS JTP 3.3.4)

This test is necessary to qualify candidate coatings for use on substrates that will be exposed to various operational fluids which could promote corrosion, including but not limited to cleaning compounds, paint removers, and deicing solutions. Test methodology is outlined in Table 19 and explained further in the following paragraphs.

Test Description

Prepare and identify (by steel stamp) three 1 inch x 1 inch x 0.032 inches (or thinner) LS1 test specimens for each test fluid. A Hull Cell panel sheared to the 1 inch x 1 inch dimensions provides an excellent test specimen. Coat three test specimens with the alternative coating, including any proposed conversion coatings to be used, to a minimum thickness of 0.0003 inches. In addition, coat three test specimens with Cd meeting the requirements of MIL-STD-870B. Clean and dry the test specimens and then store a minimum of 16 hours in a desiccator over a suitable desiccant. Following desiccation, weigh each specimen to the nearest milligram (0.001 grams). Use caution when handling specimens as coatings can be soft and easily abraded.

Test the specimens IAW ASTM F 483 (*Total Immersion Corrosion Test for Aircraft Maintenance Chemicals*) except that immersion shall be for seven (7) days. Remove the specimens from the test fluid, clean using a suitable solvent, and dry in a desiccator for at least 16 hours. Reweigh the specimens after desiccation. Analyze the test fluids for any solubilized metals that come off specimens during immersion. Rate the appearance of the exposed coupons using the criteria described in ASTM F 1110 (*Sandwich Corrosion Test*).

Immersion test fluids are listed below. It is important to note that the acceptability of vendor products is to be verified at the time of testing. Those vendor products should be identified in the test report.

- Reagent water (ASTM D 1193)
- Three parts by volume propylene glycol: one part distilled water
- Synthetic sea water (ASTM D 1141)
- Aircraft Deicing/Anti-Icing Fluid (SAE AMS 1424 / 1435)
- Runway deicing fluid (SAE AMS 1435)
- Cleaning compound, Parts Washer (MIL-C-29602)
- Cleaning compound, Aerospace Equipment (MIL-PRF-87937 Type I, Type II)
- Paint remover (MIL-R-81294)
- Paint remover (TT-R-2918 Type I (Turco 6813E))
- Paint remover (MIL-PRF-87978 Type I or equivalent)
- Paint remover – peroxide based (Reference WRALC/OCALC Purchase Description, Paint Removers for Difficult to Remove Finishes)
- Wheel well cleaning compound (MIL-PRF-85570 Type V)
- Water saturated MIL-PRF-8757
- Water saturated MIL-PRF-5606

Table 19. Test Methodology for Fluid Corrosion Resistance

Parameters	Immersion in each fluid at 100°F ±2°F (unless otherwise specified) for seven days, followed by desiccation for 16 hours. Minimum plating thickness, 0.0003 in.
Type/Number of Specimens	Three coated 4130 specimens, 1 inch x 2 inch x 0.032 inch, per candidate for each test fluid.
Experimental Control Specimens	Three 4130 specimens, 1 inch x 2 inch x 0.032 inch, Cd plated, for each test fluid.
Acceptance Criteria	No coating degradation greater than that of Cd plated control specimens as determined by weight loss and appearance.
Reference Document	MIL-STD-870B, AMSQQP416

5.1.3.3.5 Corrosion Resistance of Scribed Painted Coatings (HSS JTP 3.3.5)

This test assesses the corrosion resistance of painted candidate coatings relative to LHE Cd. This test best replicates the overall coating system that is used on landing gear and other painted Cd plated components. The original JTP only required ASTM B 117 neutral salt fog testing, but per later discussion between U.S. Army ARL and NAVAIR stakeholders an agreement was reached to use the accelerated corrosion test specification GM9540P for testing approximately half of the available panels as this test is preferred by the Army corrosion group. Test methodology is summarized in Table 20.

Test Description

Apply coating to test specimens as recommended by the manufacturer. To one set of specimens apply a waterborne epoxy primer conforming to MIL-PRF-85582 Type I, Class C1 (*Primer Coatings: Epoxy, Waterborne*), and allow to dry in air for 14 days prior to testing. To another set of specimens, apply a non-chromated waterborne epoxy primer per MIL-PRF-85582 Type I Class N (At the time of JTP preparation two primers were recommended: PRC Desoto/Spraylat EWAE118 A/B Type II, Class N or Akzo Nobel 10PW22-2/ECW119). To a third set of specimens, apply a solvent borne primer conforming to MIL-PRF-23377 Type I, Class C (*Primer Coatings: Epoxy, High Solids*). Allow all primed panels to dry in air for 14 days prior to testing. Other primers may be used as determined at the time of testing.

Machine scribe each specimen from corner-to-corner forming an “X” pattern with a 0.030 - 0.060 inches wheel cutter with a “V” cut down to a depth of plating thickness plus 0.010 ± 0.001 inch. Place test specimens in a salt spray chamber operated IAW ASTM B 117 (*Standard Practice for Operating Salt Spray (Fog) Testing Apparatus*). Examine panels in the test chamber weekly and record appearance of test panels. Monitor the first appearance and progress of white and black corrosion products. Remove the specimens from the salt spray chamber after 3,000 hours exposure or when examination reveals red rust.

Rationale

This test is for comparison purposes. Performance of the candidate coatings should be equal to or better than Cd in each type of corrosion test.

Table 20. Test Methodology for Scribed Painted Corrosion Resistance

Parameters	Conduct Salt Spray test IAW ASTM B 117: 5% NaCl solution sprayed at 35°C until coating failure. Coupons racked at 15-degree angle.
Parameters, Revised per ARL / NAVAIR	Conduct Salt Spray test IAW GM9540P with three stages: Alternating salt spray cycle, hot dry cycle, and humid cycle once per day. Coupons racked at 15-degree angle.
Type/Number of Specimens	Plated 4130 steel specimens for each primer
Experimental Control Specimens	Cd plated 4130 specimens for each primer
Acceptance Criteria	Performance equal to or better than LHE-Cd. Record observations of first appearance and progression of white and black corrosion products.
Reference Document	MIL-STD-870B, AMSQQP416

Major or Unique Equipment

Salt spray chamber, ASTM B 117

Salt spray chamber, GM9540P

Scribing machine

5.1.3.4.1 Run-on and Breakaway Torque (HSS JTP 3.4.1)

This test measures the maximum torque value during the assembly of a nut on a bolt, and the torque required to initiate removal of a threaded part (breakaway torque). Equipment schematic

used for this measurement is shown in Figure 15. If the maximum locking torque is too high, the preload is low, shortening the fatigue life. If the minimum breakaway torque is too low, the nut may vibrate off during use.

Test Description

Measure the thickness of fastener coating per MIL-STD-1312 (*Fastener Test Methods, Method 12, Thickness of Metallic Coatings*). Strip any prior finishes from fasteners to bare metal. Apply candidate coating to threaded parts listed under “Test Methodology” per manufacturer’s instructions. The applied coating must be in the same thickness class as original coating.

Lubricate the nut with SAE AMS 2518 (*Thread Compound, Anti-Seize, Graphite-Petrolatum*). Install and remove nuts once at room temperature. Use torque measuring devices conforming to requirements listed in “Major or Unique Equipment.” Record maximum locking torque value during installation and minimum breakaway torque value during removal. During the installation cycle, a peak maximum locking torque may occur as the bolt thread first enters the nut crimp. This peak locking torque is not considered part of the test.

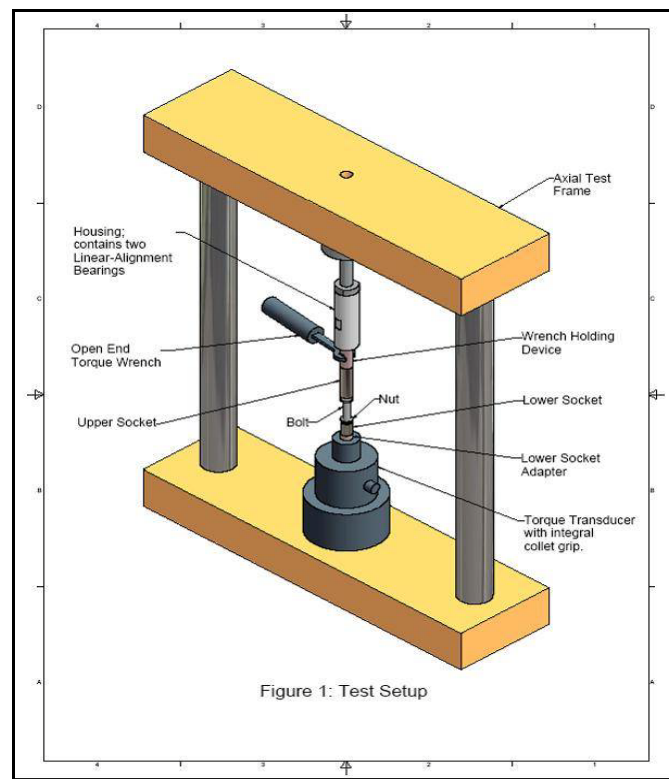


Figure 15. Maximum Locking Torque and Breakaway Torque Test Setup

Measure the maximum locking torque after two (2) complete turns (720° rotation) from the point where the top of the nut is flush with the end of the bolt. The maximum locking torque is the highest reading obtained during the third full turn (360° rotation). There shall not be any thread damage on the nut or bolt during installation when examined with the unaided eye.

Measure the breakaway torque during removal of the nut from the clamped up threaded part. Remove nut from test bolt between each cycle. Blow off any loosened particles using 60 psi of compressed air if necessary before continuing.

Measure the locking and breakaway torque values for 15 lock/breakaway cycles. After completion of the testing examine the nut and bolt for thread damage at ten times (10X) magnification. Test methodology is summarized in Table 21.

Rationale

This test is necessary to qualify candidate coatings for use on threaded parts, in order to determine the maximum locking torque and minimum breakaway torque of threaded parts. The acceptance criteria are in conformance with NASM25027.

Table 21. Test Methodology for Run-on and Breakaway Torque

Parameters	Using an adequate torque wrench, the locking and breakaway torque of fasteners was measured for 15 lock/breakaway cycles. Fasteners were also examined for damage.
Type/Number of Specimens	Test Bolts/Nuts: NASM21250-06032/ NAS1804-6 (alloy steel, candidate coated), Five specimens Test Bolts/Nuts: NASM21250-10032/ NAS1804-10 (alloy steel, candidate coated), Five specimens
Experimental Control Specimens	Test Bolts/Nuts: NASM21250-06032/ NAS1804-6 (alloy steel, Cd plated), Five specimens Test Bolts/Nuts: NASM21250-10032/ NAS1804-10 (alloy steel, Cd plated), Five specimens
Acceptance Criteria	During installation, the maximum Locking Torque shall not exceed 80 in-lb for -06032 and 300 in-lb for -10032. During removal, the minimum breakaway torque shall not be less than 9.5 in-lb for -06032 and 32 in-lb for -10032 (values from NASM25027). After 15 cycles locking torque test, nut and bolt threads shall remain in serviceable condition: no thread peel, missing segments, cracks, galling, or splits when examined at 10 times magnification; thread peel, missing segments, cracks, galling, or splits are unacceptable.
Reference Document	MIL-STD-870B, AMSQQP416

Major or Unique Equipment

Torque measuring devices shall be capable of meeting the performance requirements of and calibrated in both directions IAW MIL-T-26639 (*Tester, Torque Wrench*, issued March 14, 1969) except the torque-measuring device shall be accurate to + 4% of the indicated readings. Select torque wrenches so that the operating range of the anticipated torque values stays within 20% to 90% of the torque wrench capacity. If the torque values are not within these limits, repeat the test with the appropriate torque wrench.

5.1.3.4.2 Torque-Tension (HSS JTP 3.4.2)

This test measures the torque and load values during installation of threaded parts to achieve specified loading force. Torque-tension is measured with SAE AMS 2518 (*Thread Compound, Anti-Seize, Graphite-Petrolatum*, revised July 2001).

Test Description

Apply coating to threaded parts listed under “Test Methodology” as recommended by the manufacturer. Measure the thickness of fastener coating per MIL-STD-1312 (*Fastener Test Methods, Method 12, Thickness of Metallic Coatings*, dated July 26, 1985). The applied coating must be in the same thickness class as original coating. A schematic diagram of a generic test fixture used to measure torque-tension is shown in Figure 16.

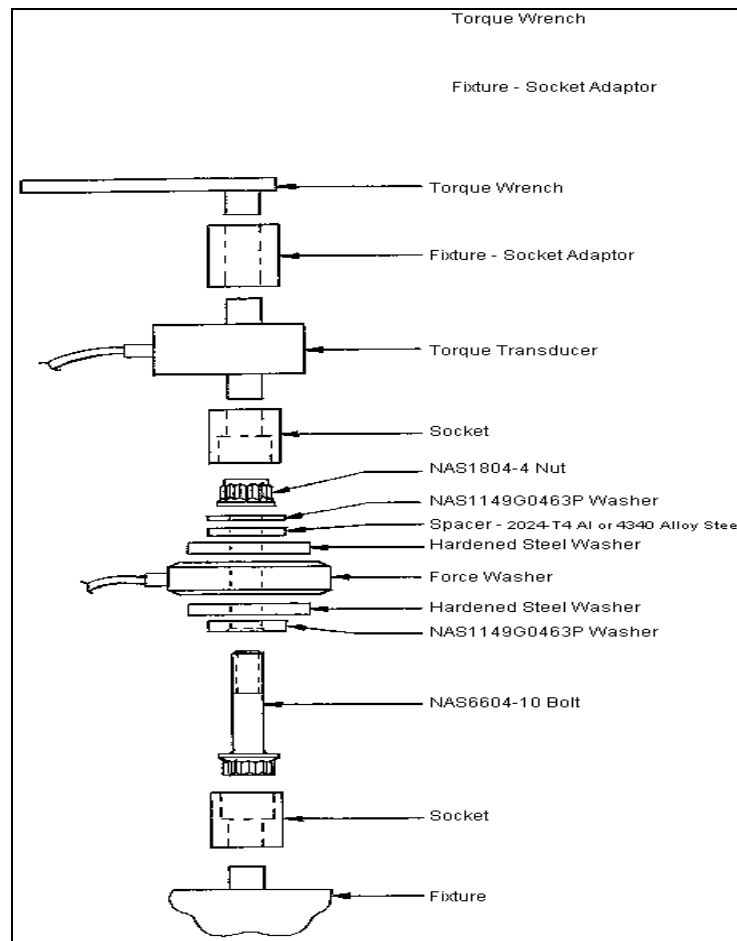


Figure 16. Schematic Torque-Tension Test Fixture

Other fixtures are acceptable provided they have the same precision. Assemble the test fixture using appropriate components sized for the test bolt and nut. Test candidate fasteners using a thread treatment complying to SAE AMS 2518 (*Thread Compound, Anti-Seize, Graphite-Petrolatum*, revised July 2001).

The test is conducted by torquing the nut. Assemble the nut onto the bolt so that a minimum of one complete thread extends beyond the top of the nut. Measure the torque-tension using the recommended torque transducer and force washer and record the torque and induced load for the range of 30% to 60% of the ultimate tensile strength (UTS) of the bolt (3,435-6,870 lb for -06032 bolts, 10,239-20,478 lb for -10032 bolts).

Repeat for a total of five cycles. A test cycle includes wrenching nut onto bolt until the desired preload is achieved, then completely removing nut. Blow off any loosened particles using 60 psi of compressed air if necessary between cycles. Record the mean and standard deviation of the test results. Plot the results. Table 22 summarizes the test methodology.

Rationale

This test is a screening test necessary for comparing the torque-tension values of candidate coated threaded parts to Cd coated threaded parts.

Table 22. Test Methodology for Torque-Tension

Parameters	Room temperature (68–78°F), installation torque range of 50–75 in-lb
Type/Number of Specimens	Test Bolts/Nuts: NASM21250-06032/ NAS1804-6 (alloy steel, candidate coated, 3/8 inch), five (5) specimens Test Bolts/Nuts: NASM21250-10032/ NAS1804-10 (alloy steel, candidate coated, 5/8 inch), five (5) specimens Test Washers: NASM14155-6 or NASM14155-10 (alloy steel, candidate coated) two (2) per test specimen
Experimental Control Specimens	Test Bolts/Nuts: NASM21250-06032/ NAS1804-6 (alloy steel, Cd coated, 3/8 inch), five (5) specimens Test Bolts/Nuts: NASM21250-10032/ NAS1804-10 (alloy steel, Cd coated, 5/8 inch), five (5) specimens Test Washers: NASM14155-6 or NASM14155-10 (alloy steel, Cd coated) two (2) per test specimen
Acceptance Criteria	Torque-tension for candidate material is within the range for Cd plated threaded parts. Threaded part does not yield or fracture, threads do not strip.
Reference Document	MIL-STD-870B, AMSQQP416

Major or Unique Equipment

Force washer/load cell that measures clamp-up
Torque measuring devices shall be capable of meeting the performance requirements of, and calibrated IAW, MIL-T-26639 (*Tester, Torque Wrench*, issued March 14, 1969) except the torque-measuring device shall be accurate to + 4% of the indicated readings. Select torque wrenches so that the operating range

of the anticipated torque values stays within 20% to 90% of the torque wrench capacity. If the torque values are not within these limits, repeat the test with the appropriate torque wrench.

GSE Inc. Model 845 Torque-tension "suitcase" (or equivalent)

Model FT-250 force washer (load cell)

Two 0.050 inches thick hardened washers

Torque transducer, 100 in-lb capacity

Spacer, 2024-T4 aluminum or 4340 alloy steel

X-Y plotter

Data Analysis

Record values during preload and plot the resulting load versus torque. Data should be linear in the elastic range. Compare to Cd control plots. Values for candidate coating should be within the torque range given for Cd.

5.1.3.4.3 Torque-Tension for Corrosion Exposed Fasteners (HSS JTP 3.4.3)

This test evaluates the effect of corrosion exposure on removal torque and torque on reinstallation of a fastener system that has been exposed to a corrosive environment. This JTP test was not completed under this effort.

Test Description

The alternate coatings should be applied as recommended by the manufacturer, to the same thickness class as LHE Cd, to the fasteners listed in Table 23. A representative test fixture to measure torque-tension was shown in Figure 16. The test nut, bolt, and washers were tested with SAE AMS 2518 (*Thread Compound, Anti-Seize, Graphite-Petrolatum*, revised July 2001).

Assemble bolts and washers in an anodized aluminum block through holes drilled through the block. Upon assembly, lubricate the fasteners with SAE AMS 2518 (*Thread Compound, Anti-Seize, Graphite-Petrolatum*, revised July 2001).

Torque the fasteners as determined in Section 5.1.3.4.2 to apply approximately 60% of ultimate load strength for the fastener. While wrenching the nut, determine and record the torque necessary to release the load for the unexposed assembly. Re-torque the fastener to approximately 60% of ultimate load and expose test block to GM 9540P cyclic corrosion for 28 cycles or 28 days.

After exposure, measure the torque necessary to remove the nut from the assembly. Remove the washers and bolt from the assembly and measure the torque-tension values IAW Section 5.1.3.4.2 for the fastener system in the same orientation as it was in corrosion exposure. Do not apply additional SAE AMS 2518 (*Thread Compound, Anti-Seize, Graphite-Petrolatum*, revised July 2001). Compare the removal torque values for the exposed fastener system with values for the unexposed fastener system.

Major or Unique Equipment

Cyclic Corrosion chamber capable of performing the GM9540P corrosion test protocol

Force washer/load cell that measures clamp-up

Table 23. Test Methodology for Corrosion Exposed Torque-Tension

Parameters	Measure torque-tension at room temperature (68–78°F); cyclic corrosion exposure IAW GM 9540P for 28 days.
Number and Type of Specimens per Candidate	Test Bolts/Nuts: NASM21250-06032 / NAS1804-6 (alloy steel, candidate coated, 3/8”), Five (5) specimens Test Bolts/Nuts: NASM21250-10032 / NAS1804-10 (alloy steel, candidate coated, 5/8”), Five (5) specimens Test Washers: NASM14155-6 or NASM14155-10 (alloy steel, candidate coated) two (2) per test specimen AL1 test block, MIL-A-8625 Type II anodized and sealed, drilled to accommodate the test fasteners
Experimental Control Specimens	Test Bolts/Nuts: NASM21250-06032 / NAS1804-6 (alloy steel, Cd coated, 3/8”), Five (5) specimens Test Bolts/Nuts: NASM21250-10032 / NAS1804-10 (alloy steel, Cd coated, 5/8”), Five (5) specimens Test Washers: NASM14155-6 or NASM14155-10 (alloy steel, Cd coated) two (2) per test specimen AL1 test block, MIL-A-8625 Type II anodized and sealed, drilled to accommodate the test fasteners
Acceptance Criteria	Torque-tension for candidate material is within the range for Cd plated threaded parts. Removal torque and torque-tension after cyclic corrosion exposure is comparable to unexposed configuration
Reference Document	MIL-STD-870B, AMSQQP416

5.1.3.5 Fatigue

Fatigue characteristics of the candidate coatings were originally planned to be evaluated using a rotating beam methodology in Phase III of this project. This technique is favored by some groups to provide representative data more quickly and inexpensively than axial fatigue methods; furthermore, some design groups use this technique to define design allowables. Other design authorities rely more heavily on axial tension-tension or tension-compression data. Some axial fatigue testing was actually conducted during Phase II of this project under NESDI leveraged funding where both air and corrosive environments were included. See Section 5.1.4.1.4 for more detailed descriptions of the actual fatigue tests conducted.

5.1.3.5.1 Rotating Beam Fatigue (HSS JTP 3.5.1)

Rotating beam fatigue tests were not conducted in this effort since Phase III was never initiated. There were, however, axial fatigue tests conducted in air environments as part of the corrosion fatigue baseline assessments. See Section 5.1.4.1.4 for more detailed descriptions of the only fatigue tests conducted as part of this project (uniaxial tension).

5.1.3.6 Hydrogen Embrittlement & Re-embrittlement

H-E of metals is a critical performance characteristic for high-strength steels. The term “hydrogen embrittlement” refers to hydrogen dissolved in the metal and characterized by delayed brittle failure of components under stress. The hydrogen is introduced into the component during application of coatings (electroplated coatings in particular). This hydrogen can be removed by baking the component in an oven soon after coating application. The permeability of the coating to hydrogen determines the success of the bake out operation. The process by which hydrogen is introduced into a component as a result of interaction of the component or its coating with the operating environment is often referred to as “hydrogen re-embrittlement” although other terms are also used (e.g., environmentally induced cracking).

There are several methods for testing the state of “embrittlement” and “re-embrittlement” with no clear consensus as to the best method, particularly in the case of “re-embrittlement”. The method used here is the standard SLT as described in ASTM F 519 for H-E and ASTM F 1624 Incremental Step Loading Technique for H-RE. It is recommended that processes be evaluated for H-E prior to the H-RE testing. Those that can produce acceptable hardware during manufacturing can then be tested for the effects of the operating environment to cause “re-embrittlement”.

5.1.3.6.1 Hydrogen Embrittlement (HSS JTP 3.6.1)

Test Description

Hydrogen embrittlement testing was included in each test phase as a quality assurance measure. This test evaluates the potential for the coating process to cause H-E IAW ASTM F 519 (*Standard Test Method for Mechanical H-E Evaluation of Plating Processes and Service Environments E* (1998), issued May 10, 1997).

Notched Fracture Strength (NFS) Verification: The NFS for the lot of specimens shall be verified by performing a standard tensile test IAW ASTM E 8 (*Standard Test Methods for Tension Testing of Metallic Materials AASHTO No.: T68 E* (2002)) on four bare, uncoated specimens. Note: This verification will need to be done for each heat treat lot of specimens. Therefore, it is strongly advised that a quantity of specimens sufficient to complete all of the tests be obtained from a single heat lot. The NFS for each bare specimen shall be within 10 ksi of the manufacturer-reported average.

Specimen Preparation: Prepare four specimens per candidate, control and baseline IAW ASTM F 519 Type 1a.1. Mask the threaded ends of these specimens and coat them IAW the issued process, including any cleaning, post plate baking, and/or conversion coating required by the coating manufacturer. Specimens are not shot peened prior to plating. Specimens shall be baked at the same temperature and duration as the actual parts to be coated—23 hours at 375°F in this effort.

Using a 10-40 times magnification (10-40X) binocular microscope, examine the entire notch surface and especially the root area of these specimens for the presence of coating. An absence of coating in the notch area shall disqualify the specimen for use in this test. Furthermore, while uniform plating thickness is not a requirement, observed thickness variations shall be documented.

Coated Specimen Notched Fracture Strength: The effect of the application of the coating and any subsequent processing on the NFS shall be established for each candidate and the control. Perform a standard tensile test IAW ASTM E 8 (*Standard Test Methods for Tension Testing of Metallic Materials AASHTO No.: T68 E (2002)*) on four coated specimens. The NFS for each coated specimen must be within 10 ksi of the manufacturer-reported average for the bare specimens.

Specimen Testing: Table 24 summarizes the methodology for this test.



Figure 17. Hydrogen Embrittlement Test Frame at NAS Patuxent River

Table 24. Test Methodology for Hydrogen Embrittlement

Parameters	ASTM E 8, bare and coated (Baseline only) ASTM F 519: Load to 75% NFS for 200 hours. Incremental Step Load (ISL) 5% NFS steps with one (1) hour dwell to fracture.
Type/Number of Specimens	Four (4) each HS1 IAW ASTM F 519 Type 1a.1 for candidate, control and baseline
Experimental Control Specimens	IAW MIL-STD-870B Class 1 Type II (thickness at 0.0005 inches to 0.0008 inches with a supplementary Cr(VI) treatment). Baseline: Bare, un-coated Type 1a.1 bar Coated ASTM E 8 tensile bar
Acceptance Criteria	NFS of bare and coated specimens within 10 ksi of average reported by manufacturer for bare <ul style="list-style-type: none"> Four of four specimens sustain 75% NFS for 200 hours SLT without fracture. - OR - Only one of four specimens fractures in less

	than 200 hours and the remaining three sustain at least one (1) hour at 90% NFS.
Reference Document	MIL-STD-870B, AMS-QQ-P-416

Test coated specimens by the SLT to 75% of the verified NFS for 200 hours. If no fracture occurs within 200 hours, increase the load by 5% NFS and hold for one (1) hour. Continue ISL step loading to fracture. A photograph of the test equipment is shown in Figure 17. For any specimen that fractures in less than 200 hours, perform a fractographic examination to determine the fracture mode and origin location using both optical and SEM methods.

Qualification Criteria: If four out of four of the notched tensile specimens sustain 200 hours SLT without fracture, the process is acceptable. If one of the four specimens fracture in less than 200 hours and the remaining three sustain least 1-hour at 90% NFS, the process is acceptable. If two (2) or more specimens fracture at less than 200 hours, the process is considered unacceptable. If the metallurgical examination reveals a preexisting defect (i.e., a crack, grinding burns, non-metallic inclusion or other anomaly) at the origin, the specimen is null. Fracture at locations other than the notch invalidates the test.

Rationale

It is known that the application of some metallic coatings to high-strength steels such as SAE 4340 at the strength levels commonly used for landing gear can induce H-E. The JTP participants agreed that this test is necessary to qualify candidate coatings. ASTM F 519 has been the aerospace industry standard for testing for H-E since its original release in 1977. Since that time, the U.S. Air Force, the Boeing Company and the aerospace industry have typically used the Type 1a.1 specimen and SLT method. As a result there is a significant historical database for the Type 1a.1 / SLT combination.

Major or Unique Equipment

Loading device as described in ASTM E 292-01 (Standard Test Methods for Conducting Time-for-Rupture Notch Tension Tests of Materials, issued October 10, 2001).
Scanning Electron Microscope (SEM)

Data Analysis

Time to fracture

Evaluation for the presence of an inclusion or other metallographic anomaly at the origin

Evaluation of the thickness, uniformity and continuity of the coating in the notch and the notch-root

5.1.3.6.2 Hydrogen Re-Embrittlement / In-Service Embrittlement (HSS JTP 3.6.2)

Test Description

This test is for screening purposes only and follows procedures described in ASTM F 1624 (*Standard Test Method for Measurement of Hydrogen Embrittlement Threshold in Steel by the Incremental Step Loading Technique*). Comprehensive testing to evaluate the susceptibility of the coating / substrate combination to H-E failures when exposed to various substances IAW Annex 5 of ASTM F 519 should be accomplished by each user group prior to implementation. Furthermore, this test will only be performed for the candidate coatings that pass the H-E testing in Section 5.1.3.6.1.

Specimen Type: Select one of the following ASTM F 519 types: Type 1a.1, 1c or 1e. The 1c and 1e types (if used) shall be tested in bending using an ISL protocol. The 1a.1 type shall be tested in tension using an Incremental Step Load (ISL) protocol. Once selected, the same type of specimen must be used for all testing. For this effort, only 1a.1 notched round bars were used.

Notched Fracture Strength Verification: The NFS for the lot of specimens shall be verified.

If the Type 1c or 1e bend specimens are selected, the bending NFS must be verified by testing four bare, uncoated specimens IAW ASTM E 8 (*Standard Test Methods for Tension Testing of Metallic Materials AASHTO No.: T68 E (2002)*). The specimens shall be tested on the same type of test frame that will be used for the re-embrittlement testing.

NOTE: This verification will need to be done for each heat lot of specimens. Therefore, it is strongly advised that a quantity of specimens sufficient to complete all of the tests be obtained from a single heat lot. The NFS (whether tensile or bending) for each bare specimen shall be within 10 ksi of the manufacturer-reported average.

Specimen Preparation: Coat four specimens IAW the issued process, including any cleaning, post coating baking, and/or conversion coating required by the coating manufacturer. Specimens are not shot peened prior to coating. Specimens shall be baked at the same temperature and duration as anticipated for actual parts to be coated.

Using a 10-40x binocular microscope, examine the entire notch surface and especially the root area of these specimens for the presence of coating. An absence of coating in the notch area shall disqualify the specimen for use in this test. Furthermore, while uniform plating thickness is not a requirement, observed thickness variations shall be documented.

Specimen Testing: The same equipment was used in this test as for H-E testing (Figure 17). Specimens were tested using both ISL protocols shown in Table 25, the shorter duration test being utilized in Phase I while the longer duration test was utilized in Phase II. The notch and gage section shall be wetted with the test fluids listed below. The test fluids shall be maintained at ambient room temperature. The test fluid shall be introduced immediately prior to the application of load. Containment of the test fluid shall be such that only the gage section of the

specimen is exposed, not the grips of the test frame. Test methodology is summarized in Table 26. The following test fluids shall be used:

- 1 mega-ohm reagent water IAW ASTM D 1193 Type 2
- Three parts by volume propylene glycol: one part distilled water
- Synthetic sea water IAW ASTM D 1141

Upon completion of each test, record the load and time to fracture. Examine the fracture surfaces using both optical and SEM methods to determine the fracture mode and origin location.

Qualification Criteria: The candidate coating is considered acceptable if the average load and time to fracture for the candidate is greater than or equal to the control (LHE-Cd) in 1 mega ohm reagent water.

Table 25. Incremental Step Loading Protocols used in Phases I and II

Load % NFS	Phase I		Phase II	
	Dwell (h)	Σ Time (h)*	Dwell (h)	Σ Time (h)
45	24	24	150	150
50	1	25	1	151
55	1	26	1	152
60	1	27	1	153
65	1	28	1	154
70	1	29	1	155
75	1	30	1	156
80	1	31	1	157
85	1	32	1	158
90	1	33	1	159
95	1	34	1	160
100	1	35	1	161
* The sigma time column reflects total cumulative test time and the associated load at that time interval.				

Rationale

It is known that the application of some metallic coatings to high-strength steels such as SAE 4340 at the strength levels commonly used for landing gear can induce H-E. In addition, it is known that, due to the sacrificial nature of some metallic coatings, these alloys may become embrittled during exposure to certain substances which can act as electrolytes.

Table 26. Test Methodology for Hydrogen Re-Embrittlement

Parameters	ASTM F 519 Type 1a.1, 1c or 1e <ul style="list-style-type: none">• Load to 45% NFS and hold for 24 hours while wetted with the test fluid, then:• ISL using 5% NFS steps with one (1) hour dwells at each stress level until bar fractures. Test Fluids <ul style="list-style-type: none">• 1 mega-ohm reagent water IAW ASTM D 1193 Type 2• Three (3) parts by volume propylene glycol: one part distilled water• Synthetic sea water IAW ASTM D 1141
Type/Number of Specimens	Four (4) each HS1 IAW ASTM F 519 Type 1a.1 for candidate, control and test fluid
Experimental Control Specimens	LHE-Cd plated per MIL-STD-870B Class 1 Type II (thickness at 0.0005 inches to 0.0008 inches with a supplementary Cr(VI) treatment).
Acceptance Criteria	Average load and time to fracture greater than or equal to LHE-Cd in 1 mega-ohm reagent water.
Reference Document	MIL-STD-870B, AMS-QQ-P-416

The JTP participants agreed that testing of specific substances would over burden the JTP and that each individual user group prior to implementation should perform such testing. Furthermore, the JTP participants agreed that this test would provide a satisfactory comparison with LHE-Cd and indication of the susceptibility of the candidate to re-embrittlement.

Major or Unique Equipment

Loading device as described in ASTM F 519 Annex 3
Scanning Electron Microscope (SEM)

Data Analysis

ISL threshold

Evaluation for the presence of an inclusion or other metallographic anomaly at the fracture origin.

Evaluation of the thickness, uniformity and continuity of the coating in the notch.

5.1.3.7.1 Repairability (HSS JTP 3.7.1)

This test evaluates the repairability of the candidate Cd-free coatings with non-Cd repair methods. The test also evaluates the use of Cd-free coatings as the repair coating for damaged Cd-plated hardware. This test is applicable for evaluating candidate repair coatings where the repair technique is done by brush plating.

The initial qualification of the brush plate solutions requires verification of the integrity of the repair coating applied on standard specimens per requirements listed in Table 27. Repair

coatings that meet the initial qualification must meet additional qualification requirements (Table 28). Final qualification requires verifying the compatibility of the repair coatings with the substrate and the surrounding plating. The integrity of the repair coatings generated by non-Cd brush plate solutions is verified on bare substrates resembling the hardware. The compatibility of the brush plate solutions is tested through the preparation of simulated repair areas. The functionality of the repaired area can be verified either non-destructively on the repaired hardware or destructively on coupons resembling the alloy of the hardware.

Test Description

For initial qualification, apply the candidate repair coating or Cd repair coating onto the bare test specimens. The repair coating can be applied by hand or by automated brush-plating equipment for increased consistency across specimens. If applied by hand, only experienced brush-plating operators shall be utilized. After the test specimens have been coated, and conversion coated if required, proceed with the initial qualification tests.

For final qualification, apply candidate coating or Cd-coating onto the test specimens, then generate a bare area on each specimen by manually abrading or machining and abrasive blasting the coating down to the substrate.

Table 27. Initial Qualification Test Method for Brush Plating Repairs

Parameters	Apply brush plate repair by experienced operator onto standardized test specimens.
Number and Type of Specimens per Candidate Coating	<p>Candidate repair coating on bare LS1 (4130 steel) and HS1 (4340 steel) test specimens.</p> <p>Appearance: 4 inches x 6 inches 4130 specimens - 1 each*</p> <p>Adhesion: 1 inch x 4 inches 4130 specimens - 3 each</p> <p>Thickness: 1 inch x 4 inches 4130 specimens - 1 each</p> <p>Unscribed Corrosion Resistance: 4 inches x 6 inches 4130 specimens - 3 each*</p> <p>Scribed Corrosion Resistance: 4 inches x 6 inches 4130 specimens - 3 each*</p> <p>Hydrogen Embrittlement (No Bake): ASTM F 519, Type 1a.1, 4340 - 4 each</p> <p>Fatigue: IAW Section 5.1.3.5.1, Rotating Beam Fatigue using smooth SAE 4340 bars only.</p>
Experimental Control Specimens	LHE-Cd plated per MIL-STD-870B
Acceptance Criteria	Repair performance meets or exceeds performance of experimental control specimens.
Reference Document	MIL-STD-870B, AMSQQP416
	Note: * = Chromate Conversion Coated

Table 28. Final Qualification Test Method for Brush Plating Repairs

Parameters	Manually scribe and abrade coating to substrate. Brush plate repair by experienced operator.
Number and Type of Specimens per Candidate Coating	Candidate repair on candidate coated 4130 steel and Cd-coated 4130 steel (2 sets total). Appearance: 4 inches x 6 inches specimens - 1 ea* Adhesion: 1 inch x 4 inches specimens - 3 each Thickness: 1 inch x 4 inches specimens - 1 each Unscribed Corrosion Resistance: 4 inches x 6 inches specimens - 3 each* Scribed Corrosion Resistance: 4 inches x 6 inches specimens - 3 each* Paint Adhesion: 4 inches x 6 inches specimens (Primer + Topcoat) - 3 each, 3 each*
Experimental Control Specimens	LHE-Cd plated per MIL-STD-870B
Acceptance Criteria	Repair performance meets or exceeds performance of experimental control specimens.
	Note: * = Chromate Conversion Coated

Machining is preferred for specimen-to-specimen consistency. Bare areas on the specimens are then repaired by brush plating with the candidate repair material on candidate-coated and Cd-coated test specimens and with Cd repair material on Cd-coated control specimens by experienced operators. The success of the repair of any damaged metal coating on a part is heavily dependent upon the proficiency of the operator performing the repair. The repair plating may be automated for increased consistency across specimens. After the repair coating has been applied to the test specimens, and conversion coated if required, proceed with the final qualification tests.

Brush-plate repaired coatings should meet the same acceptance criteria as the original coatings for non-destructive and destructive tests described in this test plan. The non-destructive tests include workmanship, appearance, tape adhesion (for paint adhesion), and thickness. These tests can be performed on the repaired hardware or the test coupons. Destructive tests include ASTM B 117 corrosion resistance (scribed and unscribed), bend-to-break coating adhesion, ASTM F 519 H-E and rotating beam fatigue. Rotating beam fatigue was not performed on the repair coatings in this test effort. Repairability with candidate non-Cd repair coatings should be demonstrated on both the candidate coated and Cd-coated specimens for each test on either 4130 or 4340 high-strength low alloy steel.

Rationale

Techniques must be available to repair scratches, gouges, worn areas and voids in the coating to return the hardware to the original design configuration and meet all acceptance criteria of this test plan. Brush plate repair of metal coatings has been successfully used to restore mis-machined parts, scratches, gouges and worn plating or bare spots on parts to drawing requirements.

Major or Unique Equipment

Abrasive Grit Blast Booth

Brush Plating Equipment: Rectifier, anodes, appropriate plating solutions, etc.

Data Analysis

As specified in original validation tests:

- Sec. 5.1.3.1.1 Appearance (HSS JTP 3.1.1)
- Sec. 5.1.3.2.1 Bend Adhesion (HSS JTP 3.2.1)
- Sec. 5.1.3.3.1 Unscribed Salt Spray (Fog) Corrosion Resistance
- Sec. 5.1.3.3.2 Scribed Salt Spray (Fog) Corrosion Resistance
- Sec. 5.1.3.5.1 Rotating Beam Fatigue (not performed under WP-0022)
- Sec. 5.1.3.6.1 Hydrogen Embrittlement

5.1.4 U.S. Navy Added Requirements

NAVAIR has additional requirements beyond those documented as common performance requirements, as described in the following sections. The Navy add on tests did not include the magnetron sputtered aluminum coating, instead it evaluated one additional electroplated zinc-nickel coating chemistry, nominally Zn-6Ni, as a control to the LHE Zn-Ni IZ-C17 process being investigated under the WP-0022 project. As some of the Navy added test specimens were obtained from different sources than the umbrella ESTCP WP-0022 project, these details will be provided in the appropriate results subsections under Sec. 6.1.4.

5.1.4.1.1 Unscribed Cyclic SO₂ Salt Spray (Fog) Corrosion Resistance (HSS JTP 4.1.1)

This test evaluates the ability of a candidate coating to prevent corrosion of coated substrates exposed to cyclic SO₂ salt spray.

Test Description

Apply coating to test specimens. Place test specimens in a salt spray chamber operated IAW ASTM G 85 Annex 4 (*Modified Salt Spray (Fog) Testing, Cyclic SO₂ Salt Spray Test*) and use controls as specified in Table 29. Examine panels in the test chamber daily for the first week and then weekly to record performance. Monitor the first appearance and progress of white and black corrosion products. Remove the specimens from the salt spray chamber when examination reveals red rust.

Rationale

This test is necessary to qualify candidate coatings for use in environments with significant exposures to low pH (acidic) conditions such as produced by SO₂ gas.

Major or Unique Equipment

Salt spray chamber, ASTM G 85 Annex 4

Table 29. Test Methodology for Unscribed Cyclic SO₂ Corrosion Resistance

Parameters	Exposure to 5% NaCl solution and SO ₂ gas IAW ASTM G85 A4 until coating failure. Coupons racked at 15-degree angle.
Type/Number of Specimens	Three candidate plated 4130 specimens
Experimental Control Specimens	Three LHE Cd plated 4130 specimens
Acceptance Criteria	Performance equal to or better than LHE-Cd. Record observations of first appearance and progression of white and black corrosion products.
Reference Document	MIL-STD-870B, AMSQQP416

5.1.4.1.2 Scribed Cyclic SO₂ Salt Spray (Fog) Corrosion Resistance (HSS JTP 4.1.2)

This test evaluates the ability of a candidate coating to prevent corrosion of coated and scribed substrates exposed to cyclic SO₂ salt spray.

Test Description

Apply coating to test specimens. Machine each specimen from corner-to-corner forming an “X” pattern with a 0.030–0.060 inches wheel cutter with a “V” cut down to a depth of plating thickness plus 0.010 ± 0.001 inch. Place test specimens in a salt spray chamber operated IAW ASTM G 85 Annex 4 (*Modified Salt Spray (Fog) Testing, Cyclic SO₂ Salt Spray Test*) and use controls as specified in Table 30. Examine panels in the test chamber daily for the first week and then weekly to record performance. Monitor the first appearance and progress of white and black corrosion products. Remove the specimens from the salt spray chamber when examination reveals red rust.

Rationale

This test is necessary to qualify candidate coatings for use in environments with significant exposures to low pH (acidic) conditions such as produced by SO₂ gas, and where the coating has sustained in-service physical damage.

Table 30. Test Methodology for Scribed Cyclic SO₂ Corrosion Resistance

Parameters	Exposure to 5% NaCl solution and SO ₂ gas IAW ASTM G 85 Annex 4 until coating failure. Coupons racked at 15-degree angle.
Type/Number of Specimens	Three candidate plated 4130 specimens
Experimental Control Specimens	Three LHE Cd plated 4130 specimens
Acceptance Criteria	Performance equal to or better than LHE-Cd. Record observations of first appearance and progression of white and black corrosion products.
Reference Document	MIL-STD-870B, AMSQQP416

Major or Unique Equipment

Salt spray chamber, ASTM G 85 Annex 4

5.1.4.1.3 Cyclic SO₂ Salt Spray (Fog) Corrosion Resistance of Scribed Painted (HSS JTP 4.1.3)

This test assesses the corrosion resistance of painted candidate coatings. This test best replicates the overall coating system that is used on painted HSS components.

Test Description

Apply coating to test specimens as recommended by the manufacturer. To one set of specimens, apply a waterborne epoxy primer, conforming to MIL-PRF-85582 Type I, Class C1 (*Primer Coatings: Epoxy, Waterborne*), and allow to dry IAW manufacturer's directions. To another set of specimens, apply a non-chromate waterborne epoxy primer, reference MIL-PRF-85582 Type I Class N (e.g. PRC Desoto/Spraylat EWAE118 A/B Type II, Class N or Akzo Nobel 10PW22-2/ECW119) and allow the primer to dry IAW manufacturer's directions. Other primers may be used as determined at the time of testing. Apply a gloss white MIL-PRF-85285 topcoat and allow the coating system to cure for two (2) weeks.

Machine each specimen from corner-to-corner forming an "X" pattern with a 0.030–0.060 inches wheel cutter with a "V" cut down to a depth of plating thickness plus 0.010 ± 0.001 inch. Place test specimens in a salt spray chamber operated IAW ASTM G 85 Annex 4 (*Modified Salt Spray (Fog) Testing, Cyclic SO₂ Salt Spray Test*) and use controls as specified in Table 31. Examine panels in the test chamber daily for the first week and then weekly to record performance. Monitor the first appearance and progress of white and black corrosion products. Remove the specimens from the salt spray chamber when examination reveals red rust.

Rationale

This test is necessary to qualify candidate coatings for use in environments with significant exposures to low pH (acidic) conditions such as produced by SO₂ gas, and where the paint and cadmium alternative coating has sustained in-service physical damage.

Table 31. Test Methodology for Scribed Painted Cyclic SO₂ Corrosion Resistance

Parameters	Exposure to 5% NaCl solution and SO ₂ gas IAW ASTM G 85 A4 until coating failure. Coupons racked at 15-degree angle.
Type/Number of Specimens	Three candidate plated 4130 specimens for each primer and topcoat
Experimental Control Specimens	Three LHE Cd plated 4130 specimens with each primer and topcoat
Acceptance Criteria	Performance equal to or better than LHE Cd. Record observations of first appearance and progression of white and black corrosion products.
Reference Document	MIL-STD-870B, AMSQQP416

Major or Unique Equipment

Salt spray chamber, ASTM G 85 Annex 4

5.1.4.1.4 Corrosion Fatigue Test (HSS JTP 4.1.4)

Cadmium alternative coatings were tested in both air environment and 3.5% sodium chloride (NaCl) solution under constant amplitude loading to determine fatigue life using hourglass specimens prepared from high strength AISI 4340 steel. The test methodology is outlined in Table 32, followed by further description.

Test Description

Specimens: Bare and Plated Hourglass Steel Specimens

Substrate Steel: 4340, 300M, AerMet 100, Hy-Tuf, HP 9-4-30, and HP 13-8 Mo

Stress Ratio (Minimum Stress/Maximum Stress): 0.1 and -1.0

Analysis: Evaluation of SEM Fractograph after Fatigue Tests

Table 32. Test Methodology for Fatigue Testing

Parameters	Fatigue test per ASTM E 466, Stress Ratio 0.1, frequency 10Hz, Air environment and 3.5% NaCl solution (pH 7.3)
Type/Number of Specimens	Bare and Plated Hourglass Steel Specimens, Six (6) specimens per coating
Experimental Control Specimens	LHE Cd plated steel Hourglass Specimens
Acceptance Criteria	Compare fatigue performance to LHE Cd
Reference Document	ASTM E 466, ASTM E 8

A closed-loop servohydraulic mechanical test machine, Interlaken, of 90 KN (20 kip) loading capacity, was employed for the tension and fatigue tests. The standard tension test was conducted in air using the round tension specimen, following ASTM E 8, Standard Test Methods for Tension Testing of Metallic Materials. The tensile loading rate was 0.076 mm/min (0.003 in/min). The fatigue test was carried out with the hourglass specimen under stress control in cyclic tension-tension at stress ratio 0.1 and frequency 10 Hz in air as well as aqueous 3.5% NaCl solution (pH 7.3). This test followed ASTM E 466, Standard Practice for Conducting Force Controlled Constant Amplitude Axial Fatigue Tests of Metallic Materials. The effect of the coatings on the mechanical performance of the base metal is evaluated by comparison to the performance of the uncoated base metal. To show that the coating does not negatively impact the mechanical performance of the base metal, the coated specimens must perform equivalent to or better than the uncoated specimens.

5.1.4.3 Stress Corrosion Cracking and Susceptibility to H-E (HSS JTP 4.3)

In this effort, since the cantilever bend and double cantilever beam SCC tests are long duration tests an accelerated SCC test was conducted in a rising step load (RSL) 1000 SI-Multi-Mode Test System per the methodology listed in Table 33 [27]. These Navy funded tests were performed only with 4340 steel.

Table 33. Test Methodology for SCC Testing

Parameters	Employ Rising Step Loading technique on notched four-point bend specimens and determine threshold stress for initiation of SCC cracks in both precracked and un-precracked conditions. Conduct testing in 3.5% NaCl solution at different cathodic potentials including OCP
Type/Number of Specimens	Single edge notched bare and coated 4340 steel specimens (modified ASTM F 519 Type 1e notched square bar); Additional substrates optional: 300M, Aermet 100, Hy-Tuf, HP 9-4-30, HP 13-8Mo
Experimental Control Specimens	LHE Cd plated specimens
Acceptance Criteria	Compare to LHE Cd; confirm results are valid with SEM on fracture surfaces
Reference Document	ASTM F 519, ASTM F 1624

The test apparatus includes a bending frame, a tensile loading frame, an electrolyte reservoir with a pump for circulation, an SCE reference electrode, a platinum counter-electrode, a computer and a printer. The as-machined, as-coated (un-precracked) and precracked specimens were step-loaded until the load dropped in four-point bending under constant displacement control, while held at a given potential in aqueous 3.5% NaCl solution of pH 7.3. The load drop corresponded to the threshold stress intensity for SCC, K_{OSCC} for the as-machined and as-coated (un-precracked) specimens and K_{ISCC} for the precracked specimens. The K_{OSCC} and K_{ISCC} were calculated as a function of applied bending moment and notch depth or crack length, using the following equation.

$$K_{OSCC} \text{ or } K_{ISCC} = \sigma \sqrt{\pi a} F(a/W)$$

$$\sigma: \text{ gross stress} = 6M/bW^2$$

$$M: \text{ bending moment} = Px$$

$$P: \text{ applied load}$$

$$x: \text{ moment arm length}$$

$$b: \text{ specimen thickness}$$

$$W: \text{ specimen width}$$

$$a: \text{ notch depth or crack length}$$

$$F(a/W) = 1.122 - 1.40(a/W) + 7.33(a/W)^2 - 12.08(a/W)^3 + 14.0(a/W)^4$$

The K_{OSCC} and K_{ISCC} values, determined at the open circuit potential (see measurement determination below), are the measure of SCC resistance of as-machined or as-coated (un-precracked) and precracked specimens, respectively, under free corrosion condition. In this investigation, K_{OSCC} was defined especially for the coated specimens, which should not be precracked. To show that the coating does not negatively impact the performance of the base metal with regards to SCC, the coated specimens must perform equivalent to or better than the uncoated specimens.

Open Circuit Potential Measurement: Open circuit potential (OCP) is an electrochemical parameter of corrosion resistance which is measurable in a corrosion cell, consisting of a specimen electrode and a reference electrode (saturated calomel electrode (SCE)) in an

electrolyte. The OCP provides conditions for use in SCC testing. In this investigation, the specimen electrode was a rectangular flat sheet, 38 x 7 x 1 mm, bare or coated. The specimen surface was coated with Stop-Off Lacquer, except for a 5 x 7 mm area on one face. This area became the working electrode in the test electrolyte, aqueous 3.5% NaCl solution of pH 7.3. The specimen and reference electrodes were connected to the ground terminals of an electrometer, and the change of electrode potential with time was recorded in reference to the SCE. The electrode potential, stabilized after 24 hours, was taken as the OCP.

5.1.5 U.S. Air Force Added Requirements

The U.S. Air Force, specifically Hill Air Force Base, has additional test requirements beyond the common performance requirements documented in the JTP.

5.1.5.1 Acceptance criteria for Torque Tension Test (Section 5.1.3.4.2)

The acceptance criteria specified by the U.S. Air Force (HAFB) for coatings to be used on wheel tie bolts is that the torque values for the required tension is within the limits stamped on the applicable wheel.

5.1.6 U.S. Army Added Requirements

The U.S. Army Aviation and Missile Command (AMCOM) has additional requirements beyond the common performance requirements documented in the JTP.

5.1.6.1 AMCOM In-Service Hydrogen Re-Embrittlement/Stress Corrosion Cracking Test Plan

Test plan for the evaluation of in-service Hydrogen Re-Embrittlement (HRE)/ Stress Corrosion Cracking (SCC) using cyclic corrosion testing per GM9540P:

- a. Use Type Id C-ring specimens IAW ASTM F 519. All of the test specimens should be made from the same heat of AISI 4340. Sensitivity tests per ASTM F 519 should be conducted for the heat.
- b. Ten (10) specimens for each of the following conditions will be evaluated.
 - i. LHE-Cd, Type II (chromate seal), Class 2 (0.0003 inches, minimum) per SAE-AMS-QQ-P-416A using MIL-STD-870B.
 - ii. LHE-Cd, Type I (as plated), Class 2 (0.0003 inches, minimum) per SAE AMS-QQ-P-416A using MIL-STD-870B.
 - iii. Bare 4340 substrate.
 - iv. Each Cd alternative with and without a seal as applicable.
 - v. Note: The coating thickness of Cd and Cd alternatives shall be between 0.0003 and 0.0005 inches.
 - vi. The coating thickness should be measured non-destructively before testing and recorded for use in data analysis upon completion of testing.
- c. Load specimens to 65% of notched bend fracture load using an appropriate test fixture making sure that no galvanic contributions exist in the assembly. The specimens should be loaded

immediately prior to corrosion testing. The bare steel substrate should be degreased with a non-embrittling solvent immediately prior to loading.

- d. Subject the loaded assemblies to cyclic corrosion test per GM9540P until failure. The test assembly should be arranged such that the loading bolt is vertical in the test chamber. Condensate should not be allowed to collect in the notch. Record the time to failure to the nearest corrosion cycle. The test assembly should be secured so that the test specimen will not drop to the bottom of the test chamber upon failure. The test assembly could be secured by carefully screwing the loading bolt into a threaded non-conductive rack.
- e. Calculate the standard deviation and the mean for the number of cycles to failure for each test condition.
- f. Establish pass/fail criteria based on the time to failure for the Cd-plated test specimens.
- g. Generate a test report per ASTM F 519 including a microstructural analysis of failed coupons and color photographs of the test specimens.

5.2 FIELD TESTING RESULTS

Two of the cadmium alternative coatings being evaluated here were being demonstrated to varying degrees during the course of this project via U.S. Air Force lead; as a result, this project was not tasked with a field demonstration of any one particular coating. These other relevant demonstration efforts will be briefly discussed as permitted in Section 8, Implementation Issues.

6.0 PERFORMANCE ASSESSMENT

6.1 JTP TEST RESULTS AND DISCUSSION

Although this project was conducted in two distinct phases and results have already been reported as individual JTR's for both Phases I and Phase II, the results will be reproduced here with appropriate modifications and further explanation where necessary; revised conclusions will be drawn now that a greater volume of data is available. Tables 34 and 35 provide top level performance metrics for each candidate coating (primary and repair, respectively) as broken out by performance parameter, listed by original JTP section number where applicable. Note that original JTP section numbers are preceded by 6.1 in this section (i.e. JTP Sec. 3.1.1 results are listed in Sec. 6.1.3.1.1 in this report). The ratings in the tables provide, at a minimum, Pass/Fail results according to the agreed-upon JCAT success criteria for each test; however, more detailed performance comparisons are made where applicable. For example, qualitative ranking numbers were provided when relative coating performance followed in a certain order. This was done to ascertain more subtle differences among the alternative coatings. A 'Pass' rating typically indicates performance equivalent or better than cadmium. In a couple tests, cadmium clearly performed lowest and this was also noted so that the strengths as well as the weaknesses of the alternatives coatings were most clear.

Because of the unique process and coating performance attributes for each alternative investigated here, each may be of value in particular engineering applications; furthermore various design entities may apply different 'weighting factors' in interpreting each JTP test in selecting a Cd alternative coating, for example commercial versus military applications, or Navy versus Air Force applications. Therefore the next few paragraphs will summarize the coatings performance in the JTP tests, while deferring individual details to the results tables, Table 34 and 35, and the full write-up for each test in the remainder of this section.

The performance tests for hydrogen embrittlement (H-E), re-embrittlement (H-RE) and bend adhesion were the most important to the JCAT team which is why they were tested first in Phase I and, furthermore, were included in each test phase to increase confidence in reproducibility of the data. Two of the alternative coatings did not pass the H-E tests in Phase I and were not included in Phase II (Sn-Zn and Boeing 'Acidic' Zn-Ni). The other coatings did not exhibit any concern in the H-E tests (Sec. 6.1.3.6.1). With respect to H-RE, all coatings underperformed Cd except the AlumiPlate™ coating. It has been observed that IVD-Al did not pass this test although it has exhibited acceptable field performance in certain applications (perhaps due to its use with chromated primers for additional corrosion protection). The sputtered Al failed at slightly lower loads than IVD Al and was therefore listed as a 'Fail' in the H-RE test, however, more data is needed to be statistically conclusive as explained in Sec. 6.1.3.6.2. The Army 'C-ring' in-service embrittlement/SCC test turned out to be somewhat too harsh to discriminate due to short failure times for almost all the specimens. In the 'C-ring' test, AlumiPlate™ performed best with sputtered Al being second best, both performing better than the baseline LHE cadmium. The Zn-Ni and Sn-Zn coatings did not perform as well as LHE Cd (Sec. 6.1.6.1). The Navy added SCC tests showed the (thicker) AlumiPlate™ coating outperformed the other alternatives in this test.

With respect to adhesion, the sputtered Al coating performed the best when considering all three substrates tested here (AISI 4130 alloy steel, PH17-4 stainless steel and titanium alloy Ti-6Al-4V), while the electroplated coatings LHE Zn-Ni and AlumiPlate™ exhibited adhesion failure principally on the more difficult to coat titanium alloy (Sec. 6.1.3.2.1). A more detailed microscopic investigation performed during Phase I had shown that IVD-Al tended to have improved adhesion on alloy steel relative to AlumiPlate™ by examining coating residues adjacent to the fracture surfaces (Appendix C). Both vacuum based deposition processes are capable of sputter cleaning the substrate surfaces prior to deposition and therefore may exhibit improved adhesion relative to electroplating, which may require a strike plating (such as nickel) prior to coating deposition.

With respect to the accelerated corrosion performance, the acidified SO₂ salt fog test (ASTM G 85 Annex 4) results showed that cadmium performs the worst of all coatings in this study (Sec. 6.1.4.1.1 – 6.1.4.1.3), in contrast to the other corrosion tests (ASTM B 117 and GM9540) where LHE Zn-Ni performs about as good as cadmium in the painted and scribed condition, which represents most high-strength steel applications (Sec. 6.1.3.3.5). These two coatings were also very similar in the unpainted condition (Sec. 6.1.3.3.1 and 6.1.3.3.2). Corrosion performance of the aluminum coatings (AlumiPlate™, IVD-Al and sputtered Al) performed approximately in that respective order with none of them being equivalent to cadmium in the ASTM B 117 or GM9540 corrosion tests.

In the painted tests, coatings with MIL-PRF-23377 Class C2 (solvent borne epoxy primer) tended to perform best compared to other primers. Typically solvent borne primers are specified for high-strength steel components as opposed to those which might be thinned with water.

The AlumiPlate™ coating was best in the fluid corrosion resistance tests by a wide margin, with the other two aluminum coatings next best. LHE Zn-Ni and LHE Cd were very similar in this test, being less resistant primarily to a subset of fluids (the paint removers) as described in Sec. 6.1.3.3.4.

Insufficient tensile specimens were run in this project (inclusive of Phase II) to statistically quantify fatigue performance differences for these coatings although trends suggested that fatigue and corrosion fatigue debits appeared to be slightly greater for Zn-Ni coatings than the aluminum coatings (Sec. 6.1.4.1.4). Depending on individual component design fatigue performance of the Cd or Cd alternative coating may or may not be weighted heavily.

With respect to threaded applications, all cadmium alternative coatings passed the run-on and breakaway torque requirements (Sec. 6.1.3.4.1) although none passed the torque-tension requirements (Sec. 6.1.3.4.2) using the anti-seize thread lubricant specified in the JTP (SAE AMS 2518, graphite-petrolatum) due to excessive variability upon repeated insertion and removal of the fasteners. Torque-tension is arguably more important than run-on and breakaway torque values since it governs application of the proper loading forces to a bolted joint.

Table 34. JTP Performance Objective Ratings for Primary Candidate Cadmium Alternative Coatings (Note: Phase II Downselects appear as column headers; coatings removed after Phase I are listed as applicable)

Performance Objective (by JTP Section Number)	LHE Cadmium	LHE Zn-Ni (IZ-C17)	IVD-Al	Sputtered Al	AlumiPlate
3.1.1 Appearance	--	Pass (1)	Pass	Pass	Pass
3.1.2 Throwing Power / Alloy Uniformity	--	Pass	Al Baseline / Pass	--	Fail
3.1.3 Strippability (tensile specimens)	--	Pass	Pass	Pass	Pass
3.1.3 Strippability (bend specimens)	--	Fail (2)	--	--	Pass
3.1.4 Electrochemical Galvanic Potential (OCP only)	--	Pass	Pass	Pass	Pass
3.2.1 Bend Adhesion – Phase I	--	Pass	Fail	Pass	Pass
3.2.1 Bend Adhesion – Phase II		Steel: Pass CRES: Pass Ti: Fail	Steel: Pass CRES: Pass Ti: Fail	Steel: Pass CRES: Pass Ti: Pass	Steel: Pass CRES: Pass Ti: Fail
3.2.2 Wet Tape Paint Adhesion	--	Pass (3)	Pass	Pass	Pass
3.3.1 Corrosion, Unscribed	--	Pass	Fail	Fail	Pass
3.3.2 Corrosion, Scribed	--	Pass	Fail	Fail	Pass
3.3.3 Galvanic Corrosion - ASTM B 117		Fail	Pass	Pass	Pass
- ASTM G 85 Annex 5	--	Pass	Pass	Pass	Pass
3.3.4 Fluid Corrosion Resistance	--	Pass	Pass	Pass	Best
3.3.5 Painted, Scribed Corrosion – B 117	Best	2 nd Best	5 th	4 th	3 rd Best
- GM9540P	Pass	Pass	Fail	Fail	Pass
3.4.1 Run-on / Breakaway Torque – 3/8” size	(4)	Pass	Pass	Pass	Pass
-5/8 inch size		Pass	Pass	Pass	Pass
3.4.2 Torque Tension	Pass	Fail	Fail	Fail	Fail
3.4.3 Torque Tension for Corrosion Exposed Fasteners	--				
3.5.1 Rotating Beam Fatigue	--				
3.6.1 Hydrogen Embrittlement	--	Pass	Pass	Pass	Pass
3.6.2 Hydrogen Re-Embrittlement (HRE) (in reagent water)	--	Fail (but > IVD-Al)	Fail / Al Baseline	Fail (5)	Pass (>Cad)
3.7.1 Repairability	See Next Table for Separate Results on Repair Coatings				
U.S. Navy Added Requirements					
4.1.1 Unscribed SO ₂ Corrosion	Pass (Worst)	Pass	Pass	Pass	Pass/Best
4.1.2 Scribed SO ₂ Corrosion	Pass (Worst)	Pass	Pass	Pass	Pass
4.1.3 Painted, Scribed SO ₂ Corrosion	--	Pass	Pass	Pass	Pass
4.1.4 Corrosion Fatigue Test		Slight debit	Pass	Pass	Pass
4.3 SCC and Susceptibility to H-E		Pass	Pass	--	Pass
4.4 Fatigue		Slight debit	Pass	Pass	Pass
U.S. Air Force Added Requirements					
5.1 Acceptance Criteria for Tension Test (Sec. 3.4.2)	--	Fail (6)	Fail	Fail	Fail
U.S. Army Added Requirements					
6.1 AMCOM In-Service H-RE/SCC	Pass	Fail	Fail	Pass	Pass/Best
(Note: Sn-Zn and “Acid Zn-Ni” had failed during this Phase I test)					
Notes: (1) LHE Zn-Ni displays a range of coloration across a plated panel—operator awareness training may be necessary where coating is implemented; (2) Coating failure after strip/re-coat has not been reproduced by other testers; (3) On LHE Zn-Ni panels, MIL-PRF-85582 Class C1 primer exhibited adhesion loss in only elevated temperature tests (120°F and 150°F immersions but passed the common JTP requirement of 1 day immersion at ambient; (4) All coatings including cadmium did not ‘Pass’ the minimum breakaway torque of 32 in-lb, Pass ratings were given by equivalence; (5) see Sec. 6.1.3.6.2 for detailed explanation; (6) Other thread treatments could potentially change relative torque-tension values for each coating here.					

Table 35. Performance Objective Ratings for each Repair Candidate Cadmium Alternative Coating

Performance Objective (by JTP Section Number)	Cadmium	Brush Zn-Ni	Brush Sn-Zn	Sermetel [®] 249
3.1.1 Appearance	--	Fail	Fail	Pass
Thickness	(baseline)	Fail	Pass	Fail
3.2.1 Bend Adhesion	Fail	Pass	Pass	Fail
3.2.2 Wet Tape Paint Adhesion	--	Not tested		
3.3.1 Corrosion Resistance, Unscribed	--	Pass	Fail	Fail
3.3.2 Corrosion Resistance, Scribed	--	Pass	Fail	Fail
3.6.1 Hydrogen Embrittlement	Pass	Pass	Pass	Pass
Notes:				

Of the repair coating alternatives, brush plated Zn-Ni performed best overall in all these tests except for appearance/thickness which may be improved with operator training (Sec. 6.1.3.7.1). The coating passed the bend adhesion requirements, the scribed and unscribed corrosion resistance and hydrogen embrittlement tests. Brush Sn-Zn did not pass the corrosion tests which are key requirements. The sprayed-on SermeTel[®] 249/273 coating performed the worst in the adhesion and corrosion resistance categories as shown above.

6.1.3.1.1 Appearance (HSS JTP 3.1.1)

In general, the appearance of all primary coatings was determined to be acceptable, and all candidate coatings, as well as baseline LHE Cd and IVD-Al coatings, were given a “pass” rating for appearance. An adequate appearance rating simply means that the coating looked reasonably uniform and acceptable, recognizing that none exactly duplicate the ‘olive drab’ cadmium finish frequently referenced in specifications. Cadmium plating exhibits some non-uniformity and edge effects as shown in Figure 18, which also illustrates representative panels of each candidate coating prior to testing (with chromated conversion coatings applied). The commercially pure aluminum coatings all have relatively similar appearance when coated with the same conversion coating solution, although the electroplated Al coating appears more reflective and smooth than the other aluminum coatings. The LHE Zn-Ni (IZ-C17) contains different shades or hues of coating in the as-plated condition. Detailed results documented from the visual examination of the primary coatings are presented in Table 36.

Table 36. Appearance of Primary Coatings

Coating	Appearance Results
LHE Cd (Baseline)	Coating is continuous but not uniform, showing some edge effect (Fig. 18); coating is smooth, adherent, and free from blisters, pits, excessive powder, and contamination
IVD-Al (Baseline)	Coating is continuous, uniform, smooth, adherent, and free from blisters, pits, excessive powder, and contamination
LHE Zn-Ni (IZ-C17 process)	Coating is continuous but not uniform, also containing a few spots of possible contamination; otherwise, the coating is smooth, adherent, and free from pits, blisters, and excessive powder; this coating inherently displays different hues of color which may require operator awareness training if the coating is implemented
Electroplated Al	Coating is continuous, uniform, smooth, adherent, and free from blisters, pits, excessive powder, and contamination
Sputtered Al	Coating is continuous, uniform, smooth, adherent, and free from blisters, pits, excessive powder, and contamination

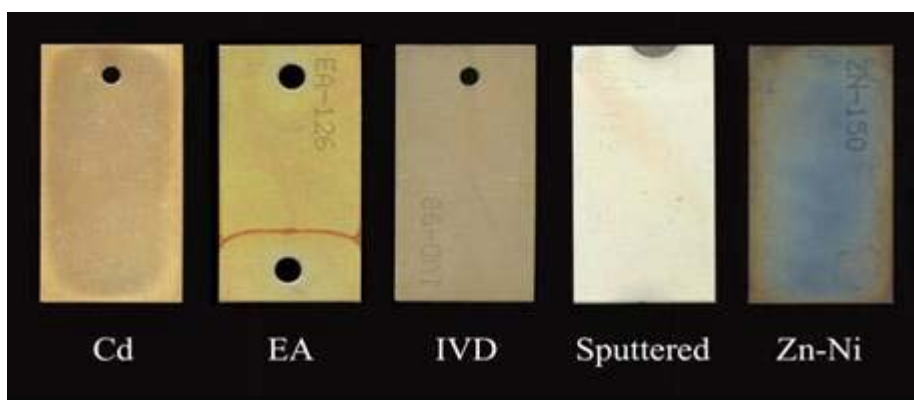


Figure 18. Representative Photo of Coated Panels Prior to Testing

6.1.3.1.2 Throwing Power and Alloy Composition Uniformity (HSS JTP 3.1.2)

Coating composition test results are listed in Tables 37 through 45. SEM was utilized to determine the composition of the coatings, using standards to calibrate the equipment. Oxygen and iron were measured for all samples in addition to the major coating constituents. Composition readings were taken at 0.5 inch intervals over the length of the panel (5 inches), at the center of the width (1.5 inches across the 3 inch width), resulting in nine readings per orientation. The first three tables list the results for LHE-Cd (Tables 37-39), the second set contains the results for electroplated Al (Tables 40-42) and the third set lists the results for LHE Zn-Ni (Tables 43-45). Throwing power samples were not prepared for IVD-Al and sputtered Al. Each table contains weight percent values of the elements in the coatings, with Reading #1 corresponding to the end of the sample closest to the open end of the fixture (composition was normalized to 100 wt.%).

Table 37. LHE Cadmium Panel Composition Results – Orientation #1

Reading #	Wt % Oxygen	Wt% Cadmium	Wt% Chromium
1	37.6%	46.2%	16.2%
2	34.6%	50.4%	15.0%
3	33.5%	51.5%	15.0%
4	32.3%	52.9%	14.8%
5	35.0%	51.1%	13.9%
6	33.6%	52.8%	13.7%
7	33.6%	53.1%	13.3%
8	33.7%	52.9%	13.4%
9	33.7%	51.2%	15.1%

Table 38. LHE Cadmium Panel Composition Results – Orientation #2

Reading #	Wt % Oxygen	Wt% Cadmium	Wt% Chromium
1	28.7%	60.5%	10.8%
2	28.0%	62.1%	9.9%
3	24.5%	65.4%	10.1%
4	26.1%	62.9%	11.0%
5	27.2%	61.1%	11.7%
6	27.9%	59.9%	12.2%
7	29.0%	58.6%	12.5%
8	29.2%	58.4%	12.5%
9	30.6%	56.8%	12.7%

Table 39. LHE Cadmium Panel Composition Results – Orientation #3

Reading #	Wt % Oxygen	Wt% Cadmium	Wt% Chromium	Wt% Iron
1	26.7%	63.4%	9.8%	0%
2	25.5%	64.1%	10.4%	0%
3	28.5%	61.0%	10.5%	0%

Reading #	Wt % Oxygen	Wt% Cadmium	Wt% Chromium	Wt% Iron
4	29.4%	59.5%	11.1%	0%
5	28.0%	56.5%	12.2%	3.3%
6	28.0%	59.1%	12.0%	1.0%
7	28.4%	56.6%	13.5%	1.5%
8	30.9%	55.1%	13.1%	1.0%
9	32.5%	53.3%	13.2%	1.0%

The orientations of the fixtures in the Cd plating bath were not provided by HAFB, so each table above is simply labeled as Orientation #1, #2, and #3. Note that these results include a substantial fraction of the Cr(VI) post-treatment which introduced chromium and oxygen peaks into the spectrum. The ratios of oxygen to Cd to chromium in the first orientation showed higher concentrations of oxygen and chromium when compared to the other two orientations which simply suggests the conversion coating is thicker. Also, the concentration of Cd varied from approximately 46% to 53%, which then dropped to 51% at the opposite edge of the panel due to potential insulation from the fixture.

Table 40. Electroplated Al Panel Composition Results – Open End in Horizontal Position

Reading #	Wt% Oxygen	Wt% Aluminum	Wt% Chromium	Wt% Iron	Wt% Nickel
1	11.8%	77.6%	5.2%	2.5%	3.0%
2	10.8%	78.7%	4.7%	2.4%	3.5%
3	10.6%	74.9%	4.8%	2.9%	6.8%
4	9.1%	72.5%	4.4%	3.2%	10.8%
5	11.7%	74.9%	5.0%	3.2%	5.2%
6	12.3%	76.8%	5.2%	3.0%	2.9%
7	12.0%	76.6%	5.6%	3.2%	2.6%
8	11.8%	76.6%	5.7%	3.0%	2.9%
9	10.8%	77.4%	4.3%	3.0%	4.5%

Table 41. Electroplated Al Panel Composition Results – Open End Facing Downward

Reading #	Wt% Oxygen	Wt% Aluminum	Wt% Chromium	Wt% Iron	Wt% Nickel
1	13.3%	76.9%	4.7%	2.6%	2.5%
2	11.5%	78.7%	4.4%	2.7%	2.6%
3	10.2%	80.0%	3.7%	2.8%	3.3%
4	9.0%	79.8%	3.8%	3.2%	4.2%
5	8.7%	81.8%	3.1%	3.3%	3.2%
6	11.1%	80.0%	3.8%	3.1%	2.0%
7	9.3%	80.4%	3.9%	3.0%	3.5%
8	10.1%	78.8%	3.9%	2.8%	4.4%
9	10.8%	77.2%	2.9%	2.8%	6.3%

Table 42. Electroplated Al Panel Composition Results – Open End Facing Upward

Reading #	Wt% Oxygen	Wt% Aluminum	Wt% Chromium	Wt% Iron	Wt% Nickel
1	9.9%	80.5%	3.9%	3.0%	2.7%
2	11.0%	79.8%	3.9%	2.9%	2.4%
3	9.4%	79.7%	3.5%	2.7%	4.8%
4	8.2%	76.8%	2.8%	3.0%	9.2%
5	8.5%	74.6%	3.5%	3.8%	9.7%
6	8.3%	78.6%	3.0%	4.4%	5.7%
7	9.4%	77.3%	3.2%	3.2%	6.9%
8	7.9%	70.9%	2.7%	5.8%	12.7%
9	3.6%	54.4%	0.9%	4.1%	37.0%

This same effect of edge insulation was also visible in the results for the second orientation. The third orientation showed the largest variance in Cd concentration over the panel, with the side of the panel that had the lower Cd concentration also displaying detectable iron concentration. This would indicate incomplete coverage of the coating over the steel substrate.

The results for the electroplated Al panels varied based upon the orientation of the fixture (Tables 40-42). The vendor stated that for these throwing power coupons they deposited the Al over a nickel strike bond layer; JCAT stakeholders had indicated a preference for not permitting nickel strikes underneath Cd alternatives in this project. Evidence of nickel signals for the AlumiPlate™ coating analyses indicates that the process did not “throw” over all areas of the panel, getting much thinner and potentially porous over the last 1.0 – 1.5 inches of the panel. When the fixture was oriented horizontally, the concentration of Al decreased by up ~5% in the center of the panel while the concentration of nickel increased. The concentrations of oxygen, chromium, and iron remained fairly consistent. For the second panel, which was oriented in the vertical position with the open end facing downward, the concentration of the elements across the panel was nearly uniform. However, there was an increase in Al concentration up to 5% at the center of the panel, an inverse of the results of the first panel. The third panel, having the open end facing upward, had a dramatic loss in Al concentration beginning at reading number 8 and extending to the end of the panel, losing almost 25% Al over this span. In addition, the nickel concentration increased by over 30% in this same measurement range.

For the LHE Zn-Ni (IZ-C17) panels, the concentration of zinc remained relatively uniform regardless of orientation while nickel concentration varied by only ± 1 -2%, depending slightly upon orientation. Traces of iron and oxygen were also detected. Nickel concentration values ranged from 11.2% on the low end to 16.1% on the high end. In the horizontal position, the nickel concentration increased across the panel (from 11.2% to 15.5%), where the samples in the vertical positions exhibited slight decreases in nickel concentration. Individual nickel concentration readings as measured here were almost entirely within the published range for this electroplating solution.

Table 43. LHE Zn-Ni Panel Composition Results – Open End Facing Node (Upward)

Reading #	Wt% Oxygen	Wt% Iron	Wt% Nickel	Wt% Zinc
1	1.6%	0.3%	15.9%	82.2%
2	1.5%	0.3%	16.1%	82.1%
3	1.9%	0.5%	15.7%	81.9%
4	1.9%	1.5%	15.6%	81.0%
5	2.1%	1.7%	15.3%	80.8%
6	1.9%	2.0%	14.1%	82.0%
7	2.0%	2.8%	14.3%	80.8%
8	2.0%	2.8%	14.0%	81.2%
9	1.5%	3.2%	13.6%	81.7%

Table 44. LHE Zn-Ni Panel Composition Results – Open End Away from Node (Downward)

Reading #	Wt% Oxygen	Wt% Iron	Wt% Nickel	Wt% Zinc
1	1.4%	0.2%	15.9%	82.5%
2	1.7%	0.3%	15.9%	82.1%
3	1.8%	0.3%	15.2%	82.7%
4	1.8%	0.8%	15.4%	82.1%
5	1.7%	1.0%	15.8%	81.6%
6	1.8%	1.3%	14.7%	82.1%
7	1.4%	1.5%	14.9%	82.2%
8	1.7%	1.7%	14.5%	82.1%
9	2.2%	2.2%	14.0%	81.6%

Table 45. LHE Zn-Ni Panel Composition Results – Open End in Horizontal Position

Reading #	Wt% Oxygen	Wt% Iron	Wt% Nickel	Wt% Zinc
1	1.7%	5.6%	11.2%	81.6%
2	2.0%	2.6%	11.9%	83.5%
3	2.2%	1.8%	13.3%	82.7%
4	2.4%	1.5%	14.3%	81.9%
5	2.2%	1.2%	15.1%	81.5%
6	2.3%	0.8%	15.4%	81.5%
7	2.1%	0.8%	15.0%	82.1%
8	2.4%	0.8%	15.2%	81.6%
9	2.6%	1.4%	15.5%	80.5%

6.1.3.1.3 Strippability (HSS JTP 3.1.3)

The first parameter which was measured for this test was the time required for chemically stripping each coating alternative. The electroplated Al coated bars which had been conversion coated at the vendor stripped in 10-15 minutes in the 130°F caustic solution. Sputtered Al bars

were received from the coater apparently without a conversion coating as corresponding strip times were much shorter (2–3 minutes) in the same solution. The LHE Zn-Ni coating strip times averaged more than an hour for what appeared visually to be full coating removal. The shortest strip time was approximately 45 minutes. When a new bath was prepared to strip the longer bend specimens (1 inch x 4 inches), pH was not adjusted far enough into the alkaline range and a lower strip rate occurred (2+ hours). This may have affected the results for bend recoat adhesion which was much lower than typical as-plated results, although if the specimens had been grit blasted prior to the re-coating process these lower results would not have been expected.

Table 46 contains a summary of the bend adhesion results after chemical strip and recoating. Baseline coatings were not included in the bend adhesion testing. In addition, sputtered Al test pieces were not returned from the vendor so there is no data for sputtered Al after recoating. As stated previously, the longer strip time required for the bend adhesion samples coated with LHE Zn-Ni may have affected the recoat adhesion properties of the coating, resulting in failures in bend adhesion.

Table 46. Summary of Strippability Test Results

Coating	Change in Bend Adhesion
LHE Cd (Baseline) – Hill AFB	Not required
IVD-Al (Baseline) – Hill AFB	Not required
IVD-Al (Baseline) – Commercial vendor	Not required
LHE Zinc-Nickel	Fail – coating failure in 1-2 bend cycles after re-coating—surface preparation uncertain with respect to grit blast)
Electroplated Al	Pass – no coating failure before substrate rupture (10-12 bend cycles)
Sputtered Al	No data

Table 47 lists the H-E results for the stripped and recoated specimens. The sputtered and electroplated Al coatings, as well as the LHE Zn-Ni coating, were able to be removed chemically from the high strength steel bars and still permit average fracture strengths of approximately 97% of the baseline notched fracture strength (NFS) for the lot of bars (without any baking step). Cd plated bars passed at an average strength of 89.4% NFS after stripping, which is slightly lower than the as-plated values determined in Phases I and II of this study (91.8% NFS and 93.7% NFS, respectively) as well as being lower than any of the re-coated alternatives.

Four additional test bars per coating were stripped and sent back to the coating suppliers for re-coating. Reworked bars were not received back for the sputtered Al coating. The specimens re-coated with electroplated Al passed with average fracture strength of 93.6% for 4 bars. Of the specimens re-coated with LHE Zn-Ni, three performed well with an average of 93.0% NFS, while the fourth failed in the threads at 13 hours (75% NFS)—thread failures are considered bad data points, but we did not process a replacement specimen. Both coatings tested achieved ‘Pass’ ratings according to the acceptance criteria.

Table 47. H-E Test Results for Chemically Stripped and Recoated Specimens

Coating	Sample #	Stripped Fracture Strength	Time to Failure*	Recoated Fracture Strength	Time to Failure*	Pass/Fail (Stripped/Recoated)
Cd-plated	1	89.7 %	203	Not tested	Not tested	Pass/(N/A)
	2	89.3 %	203			
	3	88.4 %	203			
	4	90.1 %	203			
	Avg. →	89.4 %				
LHE Zn-Ni	1	98.6 %	205	93.6 %	204	Pass/Pass
	2	99.4 %	205	75.0 %	13*	
	3	98.0 %	205	94.3 %	204	
	4	95.2 %	204	91.1%	204	
	Avg. →	97.8 %		88.5 %		
Electroplated Al	1	98.6 %	205	93.5 %	203	Pass/Pass
	2	100.0 %	205	94.2 %	203	
	3	95.6 %	205	92.0 %	203	
	4	93.6 %	204	94.5%	203	
	Avg. →	97.0 %		93.6 %		
Sputtered Al	1	95.9 %	205	Not returned	Not returned	Pass/(N/A)
	2	98.7 %	205			
	3	95.5 %	204			
	4	97.1 %	205			
	Avg. →	96.8 %				
IVD-Al		Not Tested				
* Thread failures are considered bad data points; a replacement specimen was not processed.						

6.1.3.1.4 Electrochemical Galvanic Potential (HSS JTP 3.1.4)

The full spectrum of galvanic tests outlined in the JTP was not conducted during this effort due to unforeseen circumstances. While Electrochemical Impedance Spectroscopy (EIS) and Tafel Analyses were not conducted, the Navy leveraged testing included measurements of OCP as part of SCC testing. In those tests, OCP was measured during SCC testing of the cadmium and alternative coated high strength steel notched square bars (Sec. 6.1.4.3). The OCP portion of those measurements will be reproduced here. During OCP measurement, the specimen electrode potentials displayed initial fluctuations (typical) which eventually stabilized with time. The final stabilized potential after 24 hours was recorded as the OCP. The OCP values of the bare and coated specimens are included in Table 48. Cadmium and the alternatives being tested as part of this project all had stabilized OCP values very close to -0.75V. The NESDI funded portion of this effort also included a lower nickel variety of Zn-Ni plating (listed as Zn-6Ni) which exhibited a more electronegative potential of -1.00V. To protect high-strength steel substrates the coating must maintain a potential which is neither too close to the steel nor too different. Being too close in potential a coating would not provide adequate sacrificial protection, while

being too different could more aggressively produce hydrogen at the steel surface and aggravate in-service embrittlement.

Table 48. Open Circuit Potentials (OCP) Measured on SCC Test Specimens

Coating	OCP (Volt)
Bare 4340 Steel	-0.64
AlumiPlate™	-0.75
IVD-Al	-0.74
LHE Cadmium	-0.76
Zn-6Ni*	-1.00
Zn-13Ni	-0.75
* Zn-6Ni coating was added to the NESDI funded part of this effort.	

6.1.3.2 Adhesion Test Results

6.1.3.2.1 Bend Adhesion (HSS JTP 3.2.1)

Bend adhesion testing was included in all test phases as a quality assurance measure. In this test 1"x4" steel test panels were bent back and forth until rupture of the basis metal and/or coating occurred (per ASTM B 571-97 Section 3.2). Nominally four replicates of each coating were tested. The acceptance criterion for this test was no separation (flaking, peeling, or blistering) of the coating from the basis metal at the rupture edge. Cracking was acceptable in the bend area if the coating could not be peeled back with a sharp instrument.

Phase I Bend Test Results

Several of the coatings failed this test as shown in Table 49 and Figure 19. IVD Al, brush Cd and SermeTel® 249/273 had significant flaking of the coating occur prior to substrate failure, and AlumiPlate™ had very minor peeling of the coating occur upon substrate failure. Brush ZnNi coating had some cracking near the broken edge. Based on JCAT discussion of these results adhesion failure is not typical for IVD-Al or AlumiPlate™. Representative pieces of tested IVD-Al and AlumiPlate™ panels were examined with a scanning electron microscope. Prior to examination a razor blade was used to gently lift back the AlumiPlate™ coating that had already started to peel. Elemental analysis using energy dispersive x-ray spectrometer verified that in both cases the aluminum layer separated from the steel panel, as opposed to the chromate conversion coating layer separating from the aluminum layer. The full analysis report is located in Appendix C. Brush Cd coating flaked off from the panel during the first bend cycle on all four panels. It was stated during the test results discussion that poor brush Cd adhesion is not uncommon, and that an electrocleaning step during surface preparation may promote better adhesion. SermeTel® 249/273 also failed during the first bend cycle. It was suggested during the test results discussion that a tape adhesion test may be more appropriate than bend adhesion because this product is more similar to paint than electroplating. Tape adhesion test for brush cadmium is typically only performed as a field-level adhesion check, not for qualification of new coatings. One stakeholder noted experiencing adhesion problems when using a 200 s series

SermeTel[®] product on a landing gear axle ID. Root causes of poor adhesion seen in this test were not pursued.

Table 49. Bend Adhesion Test Results – Phase I

Coating	Pass/Fail	Comments
LHE Cadmium (Baseline)	Pass	
IVD-Al(Baseline) – Hill AFB	Fail	Significant flaking/peeling
AlumiPlate [™]	Pass	Very minor peeling at broken edge
Zn-Ni (Boeing, acidic)	Pass	
Sputtered Aluminum	Pass	
LHE Zinc-Nickel	Pass	
Sn-Zn	Pass	
Repair Coatings		
Brush LHE Cadmium	Fail	Significant flaking/peeling
Brush Zn-Ni	Pass	
Brush Sn-Zn	Pass	
SermeTel [®] 249/273	Fail	Significant flaking/peeling/blistering

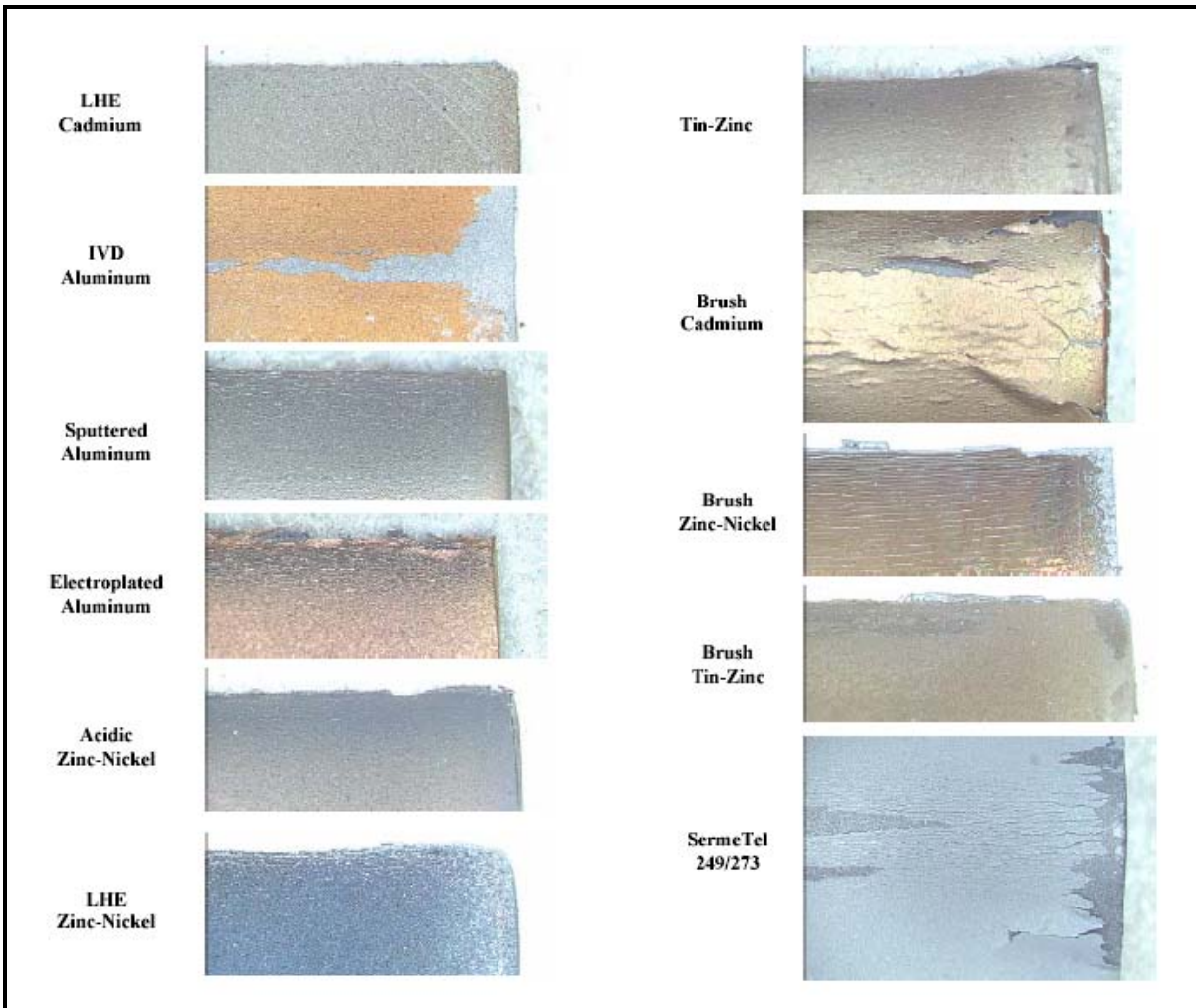


Figure 19. Photographs of Bend Adhesion Panels (Phase I)

Bend Adhesion Test Results – Phase II

Table 50 lists the results of bend adhesion testing. Alternative coatings failed strictly on the Ti-6Al-4V substrate material. Following the table, Figures 20 and 21 contain SEM micrographs and optical photographs, respectively, of the failure modes.

Table 50. Bend Adhesion Test Results

Coating	4130 steel substrate	17-4 PH stainless substrate	Ti-6Al-4V substrate
LHE Cd (Baseline) – Hill AFB	No data	Pass; 3 cycles to substrate fracture	Pass; no fracture
IVD-Al(Baseline) – Hill AFB	No data	No data	No data
LHE Zinc-Nickel	Pass – cracking of coating up to 3/8 inch; 16-18 cycles to substrate fracture	Pass – no cracking or defect; 3 cycles to substrate fracture	Fail – during 1 st bend cycle; spalling beyond 3/8 inch

Electroplated Al	Pass – cracking of coating up to 1/8 inch; 16-18 cycles to substrate fracture	Pass - no cracking or defect; 3-4 cycles to substrate fracture	Fail – edge buckling to ½ inch; 6 cycles to substrate fracture
Sputtered Al	Pass – no cracking or defect; 13-14 cycles to substrate fracture	Pass - no cracking or defect; 3 cycles to substrate fracture	Pass - no cracking or defect; 3-6 cycles to substrate fracture

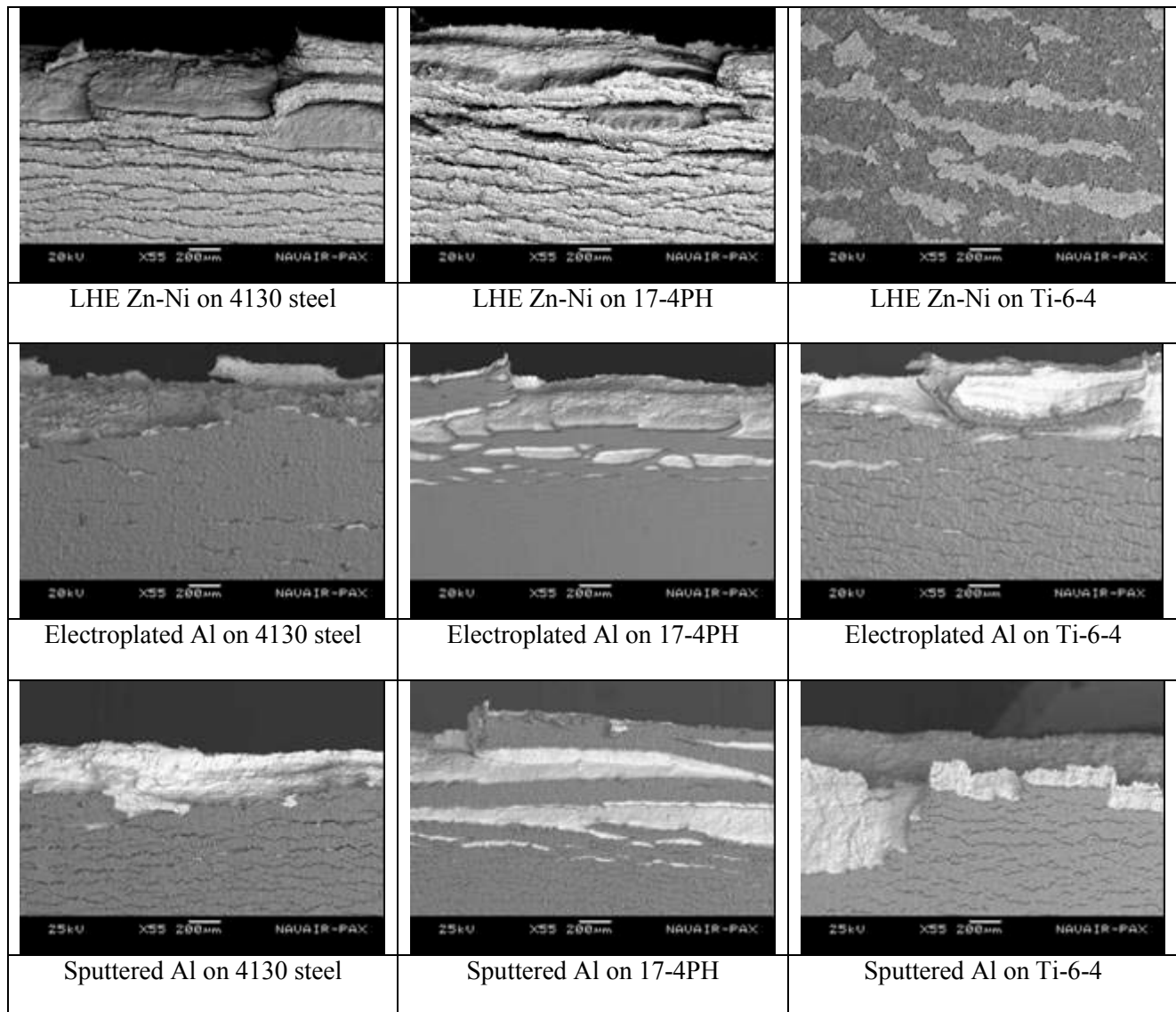


Figure 20. Low Magnification SEM Images of Bend Samples (55X)

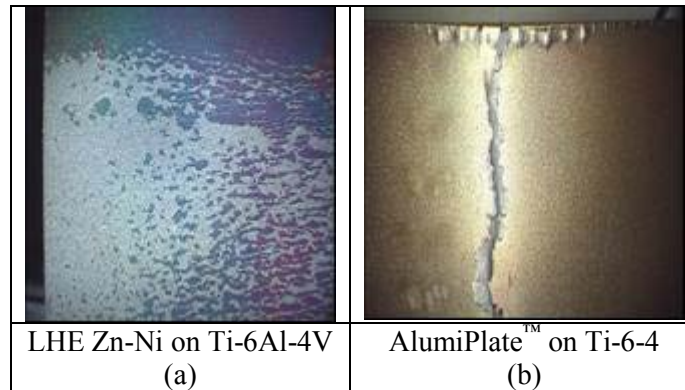


Figure 21. Optical Images of Failure Modes of Bend Adhesion Specimens

The cadmium alternative coatings tested generally displayed good adhesion to each substrate as described in Table 50 and shown in Figure 20. The only two failures noted were on titanium substrates where both LHE Zn-Ni and AlumiPlate™ coatings failed (Fig. 21). The sputtered Al coating showed the best adhesion to titanium. It should be noted that titanium is a difficult substrate for activation when electroplating, and that the failures experienced on these substrates might be attributable to surface preparation rather than coating properties. IVD-Al was not tested here, but it is anticipated that both sputtered and IVD-Al have adhesion advantages (on metals like titanium) due to the plasma surface cleaning associated with the vacuum based coating processes.

Although through-thickness cracks appeared in the Al coatings near the fracture surface due to the severe deformation, they were not able to be lifted with a sharp blade. On Ti-6Al-4V, however, the electroplated Al coating buckled and lifted along the sides of the bend sample up to ~0.5 inch away from the fracture plane (Figure 21b). These areas could be removed with a blade. According to the manufacturer, the electroplated Al had been deposited over a nickel strike bond layer for coating on titanium. The LHE Zn-Ni plating performed well on 17-4 PH stainless steel and 4130 steel, although the plating flaked off the Ti-6Al-4V substrate before one full 90 degree bend of the substrate. The coating came off in 0.02–0.04 inch flakes, and exhibited spalling beyond 0.375 inch from the fracture plane.

Bend Adhesion Testing – Repair Coatings (Phase II)

Bend adhesion testing was also conducted in the same manner as the primary coatings, by clamping the specimen into a vice and bending the free end back and forth (one cycle) until failure of the coating or substrate occurs. The results of bend adhesion testing of the repair coatings are listed in Table 51. In addition, Figure 22 contains representative photos of the test specimens after testing. Bend adhesion test results for the repair coatings were similar to those obtained in Phase I (again only tested on low alloy 4130 steel). Brush plated Cd exhibited significant adhesion loss after 1.5 bend cycles (second tensile cycle for the coating). Application of a sharp blade easily removed most of the coating. Brush Sn-Zn performed well on all specimens. One representative panel is shown in Figure 22. The Brush Zn-Ni panels performed well on all specimens, with only slight coating removal at the edges of the specimens. The bulk

of the area did not have visually apparent cracks developing until 8-9 bend cycles, and the deposit adhered well when trying to lift it with a blade after substrate failure.

Table 51. Bend Adhesion Results for Repair Coatings

Coating	Replicate	Cycles to Failure	Comments	Pass/Fail
Brush Plated Cd	1	2-3	Significant coating adhesion loss after 1.5-3 cycles	Fail
	2	2		
	3	2-3		
Brush Plated Zn-Ni	1	13	Coating cracks at 8-9 cycles	Pass
	2	11		
	3	12		
Brush Plated Sn-Zn	1	10		Pass
	2	17		
	3	14		
Al-Ceramic (SermeTel [®] 249/273)	1	2	Coating failure across full width of 2 specimens	Fail
	2	2-3		
	3	3		

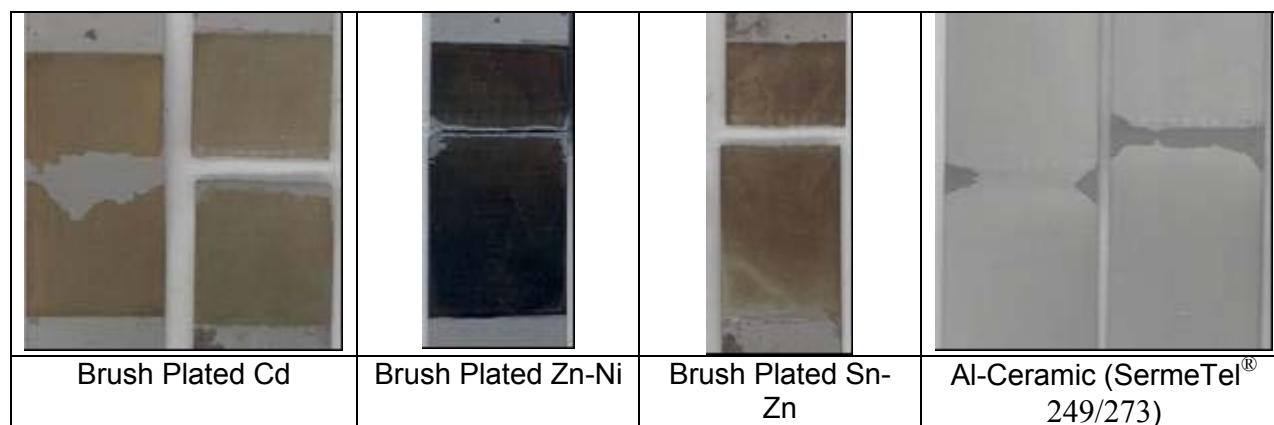


Figure 22. Representative Photos of Bend Adhesion Results for Repair Coatings

The SermeTel[®] coating exhibited adhesion failure after 2-3 cycles. Light brushing with a fingernail removed the coating across the full width of one specimen out of three, while the other two exhibited removal closer to the edges (pictured in Figure 22).

6.1.3.2.2 Wet Tape Paint Adhesion (HSS JTP 3.2.2)

This test assessed the general adequacy of paint adhesion to flat surfaces coated with the candidate coating. The test was conducted by applying and removing pressure-sensitive adhesive tape over scribes made through the primer. Elevated temperature testing conducted at 49°C and 65°C did not have acceptance criteria established as common performance requirements, however, military primer specifications typically include elevated temperature water immersion tests for the primer on conversion coated aluminum surfaces (e.g, MIL-PRF-85582 (4 days at 120°F); TT-P-2756 (7 days at 150°F)).

Table 52 lists the results of the tape adhesion test for each coating system, at each time/temperature combination. The specific tape used for this testing was 3M #250 with a nominal adhesion value of 75-80 ounces/inch. Each coating was tested on triplicate 4 inch x 6 inch steel panels for each duration test, using four adhesion test sites per panel, yielding a total of twelve values which were averaged to provide the data for each entry below. Average primer thickness values were as follows: MIL-PRF-23377 Class C2 (2.0 mil), MIL-PRF-85582 Class C1 (0.9 mil), MIL-PRF-85582 Class N (1.2 mil). Figure 23 contains a few representative photos of the tape adhesion panels.

Table 52. Averaged Wet Tape Adhesion Results

Primer application →	MIL-PRF-23377, Class C2			MIL-PRF-85582, Class C1			MIL-PRF-85582, Class N		
Test Duration →	1 day	4 days	7 days	1 day	4 days	7 days	1 day	4 days	7 days
Coating	Test Results (average of 12 measurements)								
Cd-plated (control)			5B ¹			5B ¹			5B ¹
IVD-Al (Hill AFB) ²	5	5	5	5	5	5	5	5	5
LHE Zinc-Nickel ³	4.8	4.4	4	5	1.3	0.83	5	5	5
Electroplated Al	5	5	5	5	5	5	5	5	5
Sputtered Al	5	5	5	5	5	4.7	5	5	5

¹ Cd plated panels were inadvertently topcoated with MIL-PRF-85285.

² Primer pinholes were more prevalent with IVD panels, especially with MIL-PRF-85582 C1 primer.

³ A solvent wipe prior to painting may have improved results, although compatibility with MIL-PRF-85582-N primer was excellent.

All inorganic coatings passed the JTP criterion for paint adhesion with ratings no less than 4.8 in the one day ambient temperature test. Additionally, all of the chromate post-treated Al coatings performed very well including the seven day test with typical ratings of 5.0. The LHE Zn-Ni coating exhibited slightly lower average paint adhesion values with MIL-PRF-23377 primer in the elevated temperature tests (4.0-4.4 ratings), but performed very well with the non-chromate primer MIL-PRF-85582 Class N (5.0 values).

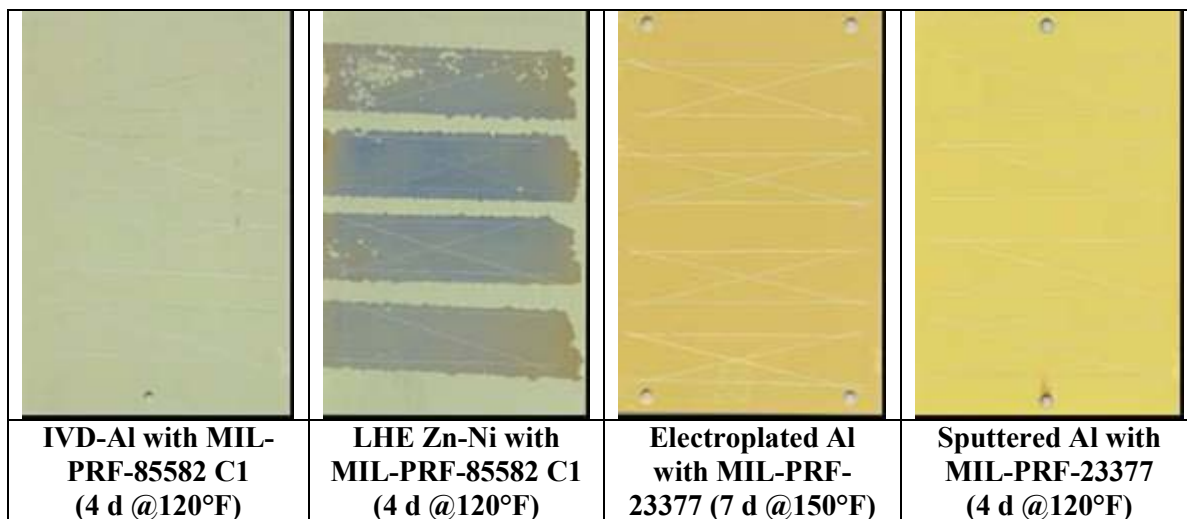


Figure 23. Representative Photos of Wet Tape Adhesion Panels

Poor adhesion was observed on the Zn-Ni with MIL-PRF-85582 Class C1, with primer blistering and substantial coating removal by the tape. All of the panels painted and tested in this study were treated the same prior to painting, as they were unwrapped from the coater's packaging the day prior to primer application. Typically, the panels were individually wrapped and sealed in zippered plastic bags.

6.1.3.3 Corrosion Resistance Results (HSS JTP 3.3)

6.1.3.3.1 Unscribed Salt Spray (Fog) Corrosion Resistance (HSS JTP 3.3.1)

This test evaluated the ability of a candidate coating to prevent corrosion of coated substrates exposed to salt spray.

Table 53 lists the results of the salt spray exposure of unscribed panels for each of the primary coatings. The table includes the time when red rust first appeared, as well as a description of the coating and rating at the end of the test for each panel tested. Panels were rated IAW ASTM D 1654. Photos of each of the coatings at the conclusion of the test are located in Figure 24, after the table.

Table 53. Corrosion Resistance Results – Unscribed Panels

Coating	Panel #	First Appearance of Corrosion/Observation	Time of Termination/Observation	Rating at Test Termination
Cd-plated	1	No rust	3000 hours/No damage	9
	2	No rust	3000 hours/No damage	9
	3	No rust	3000 hours/No damage	9
IVD-Al (baseline – Hill AFB)	1	1500 hours/ Significant rusting	1500 hours/ Excessive rusting	0
	2	2000 hours/Sacrificial coating breakdown – pinhole rust	3000 hours/ Significant rusting	0
	3	72 hours/ Significant rusting	168 hours/ Excessive rusting	0
LHE Zinc-Nickel	1	No rust	3000 hours/ Sacrificial coating breakdown/ no rust	9
	2	No rust	3000 hours/ Sacrificial coating breakdown/ no rust	9
	3	No rust	3000 hours/ Sacrificial coating breakdown/ no rust	9
Electroplated Al	1	1500 hours/ Chromate depletion and pin holes	3000 hours/ Chromate depletion and pin holes	7
	2	1500 hours/ Chromate depletion and pin holes	3000 hours/ Chromate depletion and pin holes	7
	3	1500 hours/ Chromate depletion and pin holes	3000 hours/ Chromate depletion and pin holes	7
Sputtered Al	1	500 hours/ Excessive rust	500 hours/ Excessive rust	0
	2	500 hours/ Excessive rust	500 hours/ Excessive rust	0
	3	500 hours/ Excessive rust	500 hours/ Excessive rust	0



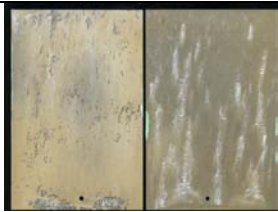
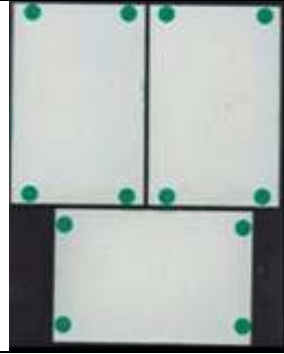
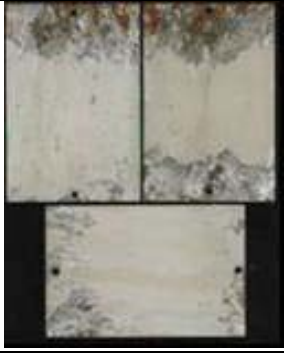

		
Cd plated (3000 hours)	LHE Zn-Ni (3000 hours)	IVD-Al (1000 hours)
		
Electroplated Al (3000 hours)	Sputtered Al (500 hours)	IVD-Al (Panel 2) (3000 hours)

Figure 24. Photos of Unscribed Corrosion Resistance Panels After Exposure

The results listed in the table show that the Cd-plated panels met the specification of showing no rust after 3000 hours salt spray exposure. LHE Zn-Ni also performed well showing breakdown of the sacrificial coating after 3000 hours exposure but no red rust formation. The electroplated Al coating began exhibiting depletion of the chromate conversion coating at 1500 hours (assumed by color change not by direct measurement) exposure along with some pinhole formation. The appearance of the panels at 3000 hours was the same, indicating that the pinholes did not progress in size during the second half of the test to result in additional red rust.

Two of three IVD-Al panels and the sputtered Al panels developed red rust and were pulled from testing prior to the completion of 3000 hours exposure. The first appearance of corrosion on the IVD-Al panels varied greatly from 72 hours to 2000 hours. IVD-Al corrosion resistance is somewhat dependent on the burnishing or coating densification process, so therefore the panel edges would be more susceptible to failure as they do not receive a controlled burnish and are even likely to be thinned excessively at the edges. Figure 24 shows two IVD-Al panels at 1000 h and also the one that survived until 3000 hours. The SAE AMS 2427 specification for IVD-Al coatings indicates that the acceptance criteria for IVD-Al corrosion resistance is 504 hours for chromate treated coating (0.0005-0.001" thickness category). One of the three IVD-Al samples tested did not meet this acceptance criterion. The sputtered Al panels were more consistent in developing excessive rust within 500 hours, terminating the test.

6.1.3.3.2 Scribed Salt Spray (Fog) Corrosion Resistance (HSS JTP 3.3.2)

This test evaluated the ability of candidate coatings to protect substrates from corrosion even when damaged through the full thickness of coating.

Table 54 lists the test results for the scribed corrosion resistance test panels, rated IAW ASTM D 1654. The time of the first appearance of corrosion is listed along with time of test termination and final rating. Photos are presented in Figure 25.

Table 54. Corrosion Resistance Results – Scribed Panels

Coating	Panel #	First Appearance of Corrosion/Observation	Time of Termination/Observation	Rating* at Test Termination
Cd-plated	1	No rust	3000 hours/No damage	9
	2	No rust	3000 hours/No damage	9
	3	No rust	3000 hours/No damage	9
IVD-Al(baseline – Hill AFB)	1	72 hours/ Excessive rusting	72 hours/ Excessive rusting	0
	2	72 hours/ Significant rusting	168 hours/ Excessive rusting	0
	3	500 hours/ One rust spot on scribe	1000 hours/ Excessive rusting	0
LHE Zinc-Nickel	1	No rust	3000 hours/ Sacrificial coating breakdown/ no rust	9
	2	No rust	3000 hours/ Sacrificial coating breakdown/ no rust	9
	3	No rust	3000 hours/ Sacrificial coating breakdown/ no rust	9
Electroplated Al	1	500 hours/ Chromate depletion and pin holes	3000 hours/ Sacrificial corrosion & rust in scribe	0
	2	1000 hours/ Chromate depletion and rust in scribe	2000 hours/ Sacrificial corrosion & significant rust	0
	3	1000 hours/ Chromate depletion and rust in scribe	2000 hours/ Sacrificial corrosion & significant rust	0
Sputtered Al	1	500 hours/ Excessive rust	500 hours/ Excessive rust	0
	2	500 hours/ Excessive rust	500 hours/ Excessive rust	0
	3	500 hours/ Excessive rust	500 hours/ Excessive rust	0
* Ratings provided IAW ASTM D 1654				

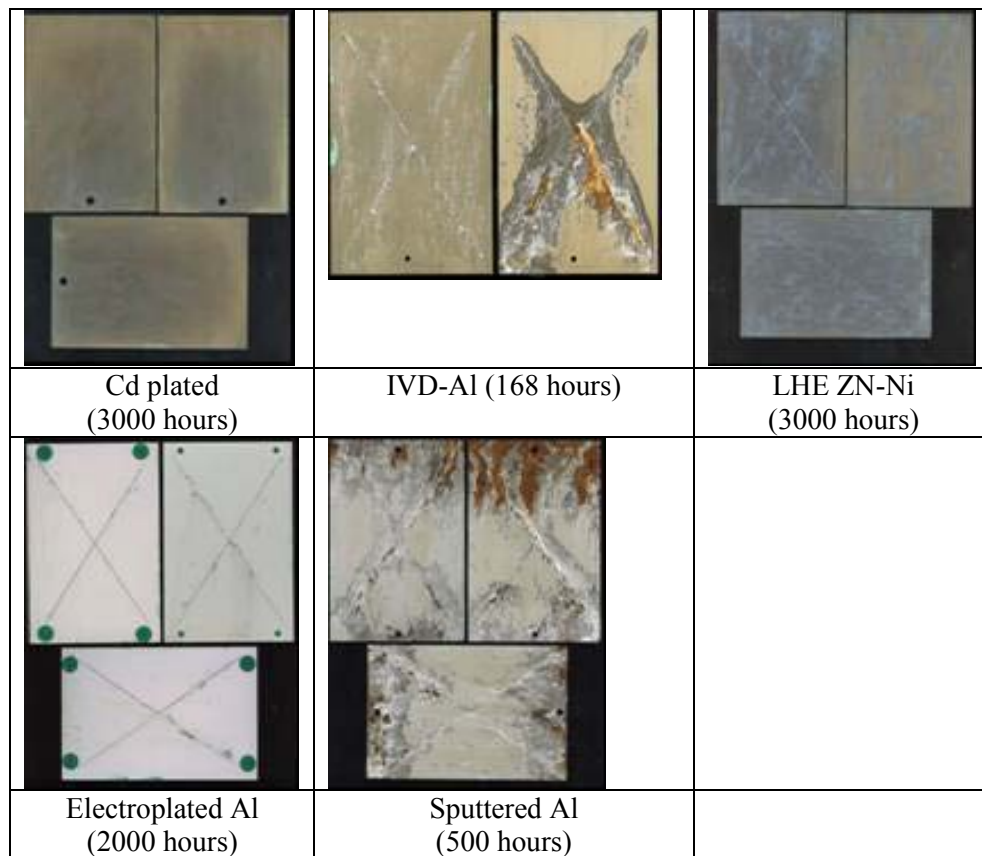


Figure 25. Photos of Scribed Corrosion Resistance Panels after Exposure

The results obtained for the scribed panels were very similar to the results for the unscribed panels. The Cd-plated panels did not exhibit any corrosion from the salt spray as expected, and LHE Zn-Ni received the next highest rating by exhibiting only some sacrificial coating breakdown but no rust even in the scribe. The electroplated Al followed, although two of three panels had to be pulled prior to 3000 hours due to significant rust. Once again, the IVD-Al and sputtered Al panels exhibited the most significant corrosion so testing was terminated at or before 1000 hours.

6.1.3.3.3 Galvanic Corrosion Resistance (HSS JTP 3.3.3)

Providing galvanic compatibility between the dissimilar metals used on high strength steel components and assemblies is a critical function of candidate coatings.

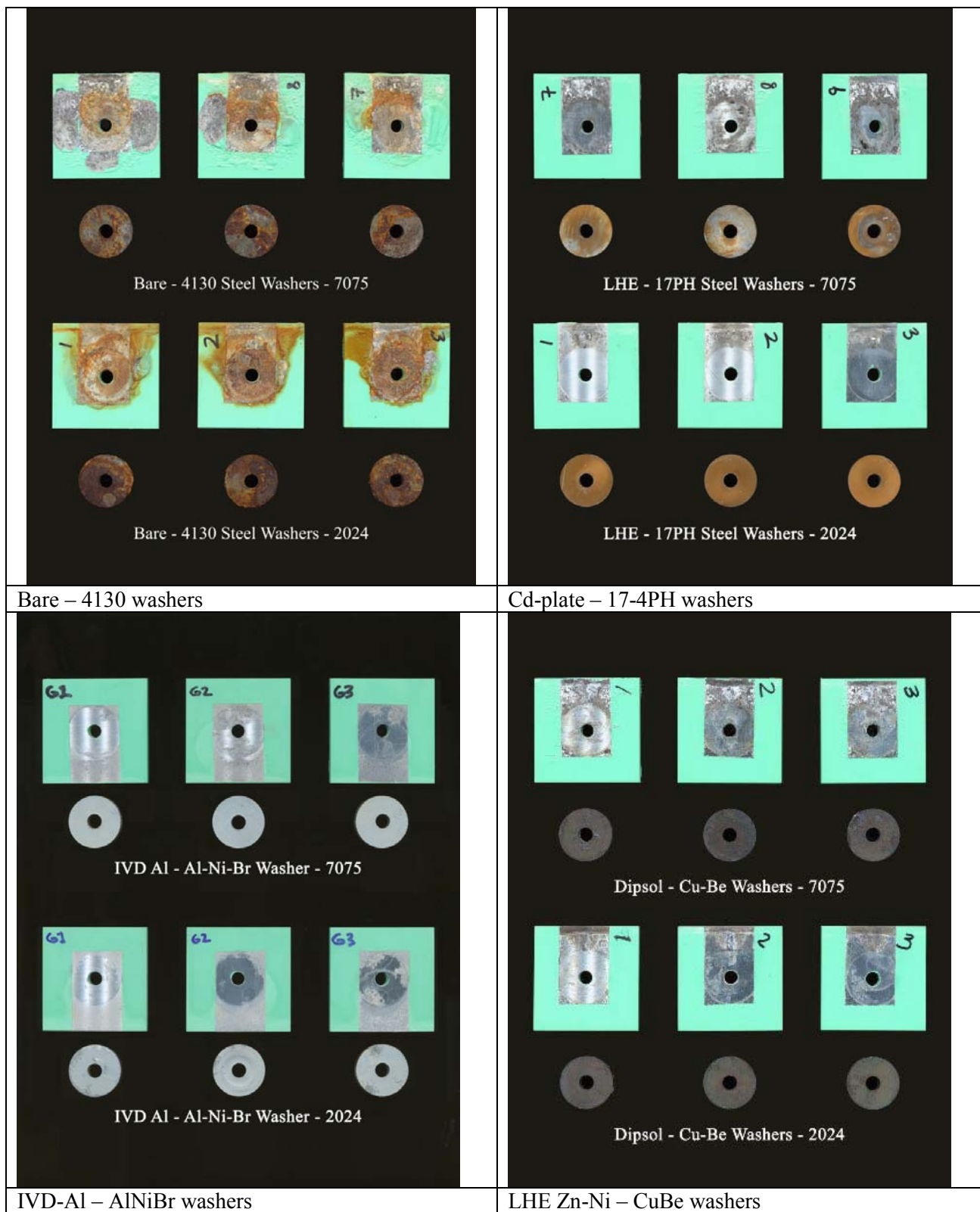
Table 55 lists the change in electrical resistivity for each coating and each substrate material, for 2024 Al and 7075 Al alloy test blocks, exposed to ASTM B 117 salt fog for 168 hours. Figure 26 contains photos of the salt spray-exposed assemblies. Table 56 lists the same information for the samples exposed to cyclic corrosion for 336 hours, followed by Figure 27, with photos of the specimens.

Both the electrical test results and images indicate that as a whole the fixtures performed better after two weeks of GM9540P cyclic corrosion exposure than one week of ASTM B 117 neutral

salt spray. As expected the bare test washer baseline had the most corrosion and exhibited very large increases in resistivity. The Cd-plated test washers had no resistivity increases in most configurations except for the 17-4PH stainless steel substrate (same observation in both the neutral salt spray and cyclic corrosion tests). The alternative coating that had the most changes in resistivity upon salt spray exposure was LHE Zn-Ni. This was followed by electroplated Al and sputtered Al, which both had small changes in resistivity for almost every test washer/test block alloy combination. The exception was electroplated Al on Cu-Be, which had no change in resistivity. All the alternative coatings exhibited little to no change in resistivity for assemblies exposed to cyclic corrosion for a period of two weeks.

Table 55. Change in Resistivity for ASTM B 117 Exposed Fixtures

Coating	Test Block Substrate	Change in Resistivity (milliohms) 3 Fixtures Each			
		4130 Washer	17-4PH Washer	Cu-Be Washer	AlNiBr Washer
Bare (no coating)	2024 Al	62.3, 114, 87.7	66.5, 6.9, 84.6	13.4, 78.5, 1.9	0, 0, 0
	7075 Al	165, 0, 39.8	38.7, 84.3, 687	0.7, 18.5, 18.6	11.8, 0, 0
Cd-plated	2024 Al	0, 0, 0	0.6, 0.4, 0.3	0, 0, 0	0, 0, 0
	7075 Al	0, 0, 0	0.2, 0.3, 0.2	0, 0, 0	0, 0, 0
IVD-Al	2024 Al	0, 0, 0	0, 0, 0.1	0, 0, 0	Infinity, 0, 0
	7075 Al	0, 0	0, 0, broke	0, 0, 0	0, 0
LHE Zn-Ni	2024 Al	0.4, 0.2, 0.4	0.9, 0.4, 0.1	5.1, 1.1, 0	Broke, 0.3, 0.1
	7075 Al	0.5, 0.4, 0.5	11600, 0.6, 0.6	5.6, 2.3, 1.7	1.1, 0.5, 0.6
Electro-plated Al	2024 Al	0.3, 0.2, 0	0, 0, 0	0, 0, 0	0.4, 0.5, 0.5
	7075 Al	0.3, 0.7, 3.6	1.0, 0.8, 0	0, 0, 0	1.0, 0.5, 0.7
Sputtered Al	2024 Al	0.3, 0, 0	0, 0, 0.2	0.1, 0, 0.1	0, 0.2, 0
	7075 Al	0, 0, 0	0, 0, 0	0, 0.5, 0	0, 0.4, 0.6



**Figure 26. Representative Photos of ASTM B 117 Salt Spray-Exposed Fixtures
(continued on next page)**

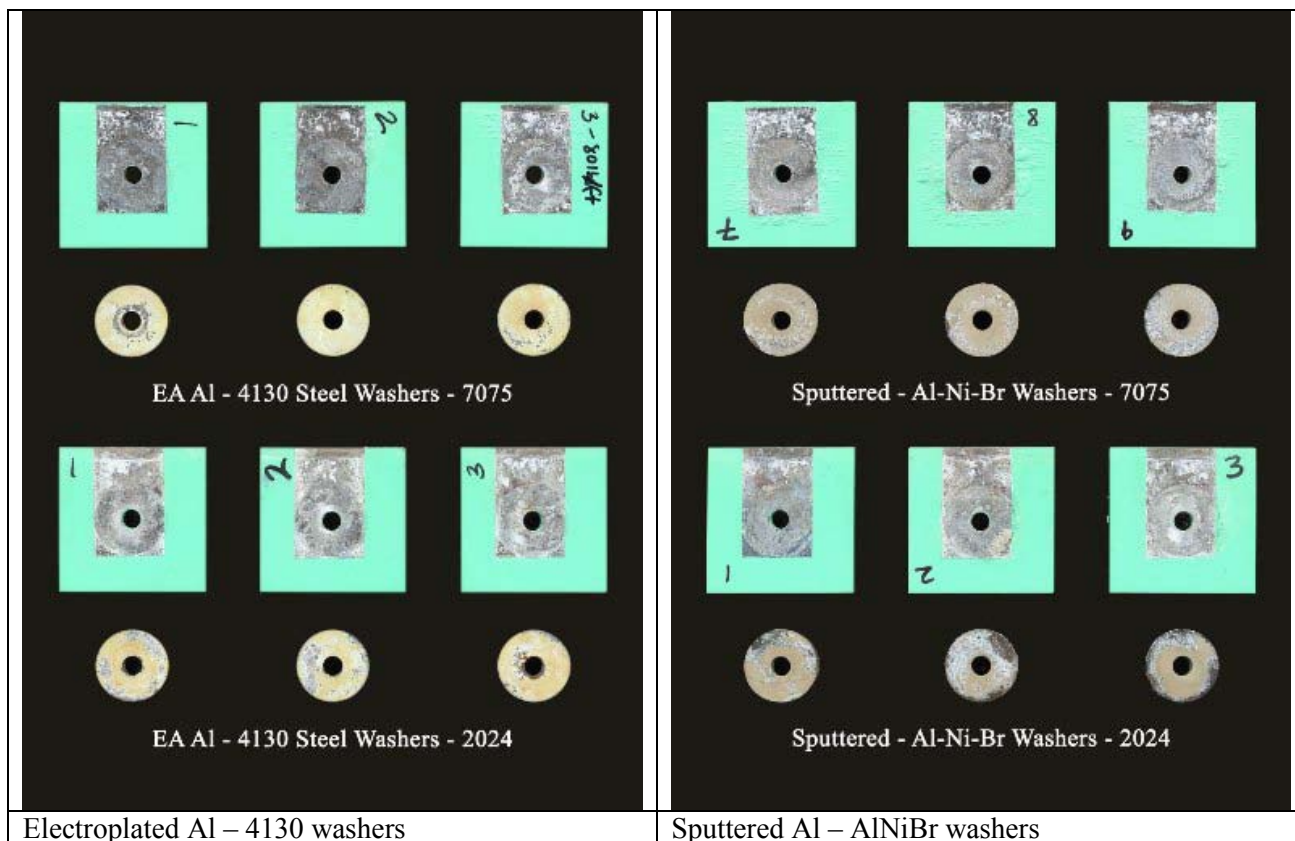
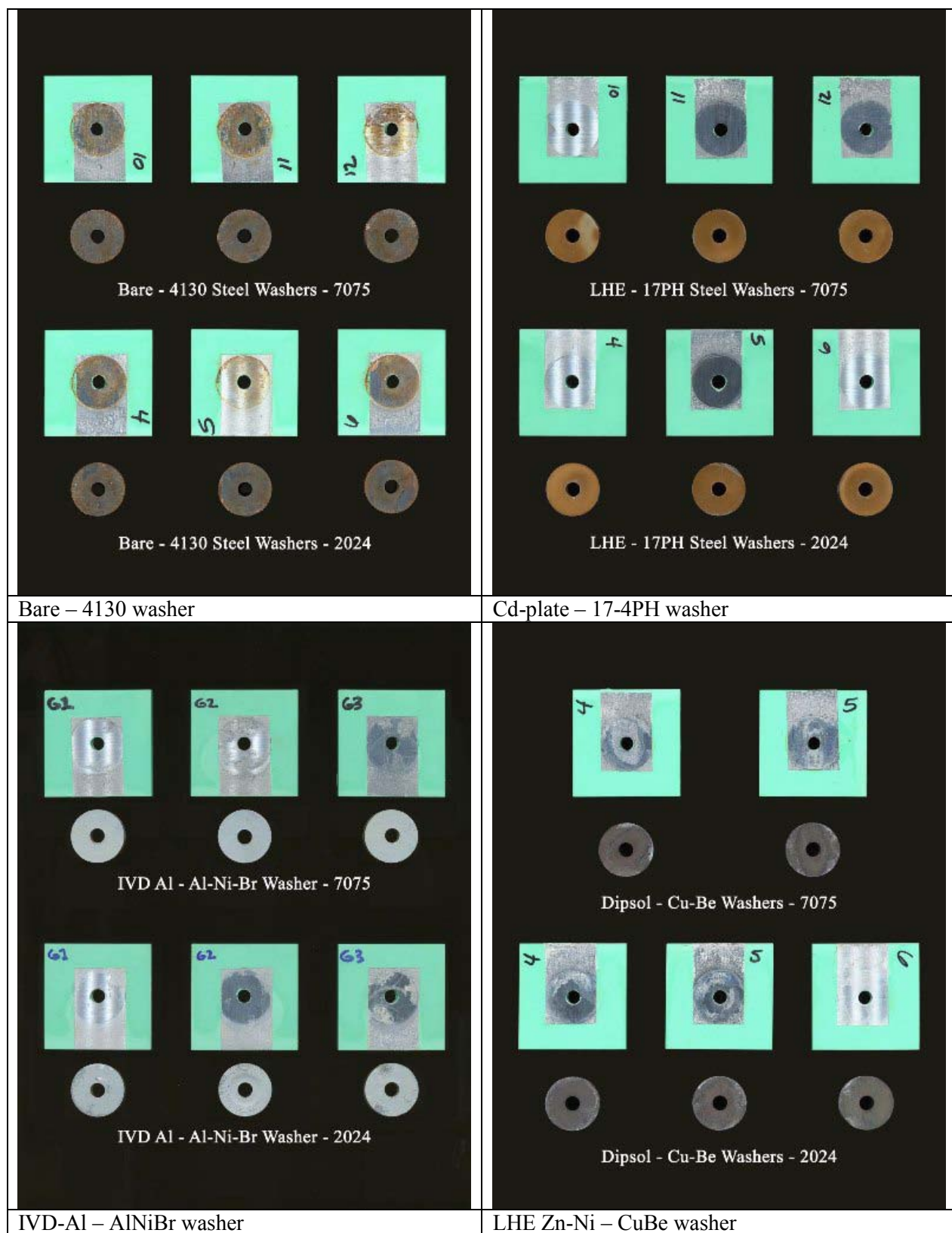


Figure 26. Representative Photos of ASTM B 117 Salt Spray-Exposed Fixtures

Table 56. Change in Resistivity for Cyclic Corrosion Exposed Fixtures

Coating	Test Block Substrate	Change in Resistivity (milliohms) 3 Fixtures Each			
		4130 Washer	17-4PH Washer	CuBe Washer	AlNiBr Washer
Bare (no coating)	2024 Al	80000, 0, 550000	5.6, 0.8, 1.4	0, 0, 0	0, 0, 0
	7075 Al	660000, 1.0, 0.8	15300, 0, 0	0, 0, 0	0, 0, 0
Cd-plated	2024 Al	0, 0, 0	0.6, 0.3, 0.2	0, 0, 0	0, 0.8, 0.6
	7075 Al	0, 0, 0	0.6, 0, 0.8	0, 0, 0	0, 0, 0
IVD-Al	2024 Al	0, 0, 0	0, 0, 0	0, 0, 0	0, 0, 0
	7075 Al	0, 0, 0	0, 0, 0	0, 0, 0	0, 0, 0
LHE Zn-Ni	2024 Al	0, 0, 0	0, 0, 0	0, 0, 0	0, 0, broke
	7075 Al	0, 0, 0.1	0, 0, 0	0, 0, broke	0, 0, 0
Electro-plated Al	2024 Al	0, 0, 0	0, 0, 0	0, 0, 0	0, 0, 0
	7075 Al	0, 0, 0	0, 0, 0	0, 0, 0	0, 0, 0
Sputtered Al	2024 Al	0, 0, 0	0, 0, 0	0, 0, 0.1	0, 0, 0
	7075 Al	0, 0, 0	0, 0, 0	0, 0, 0	0, 0, 0



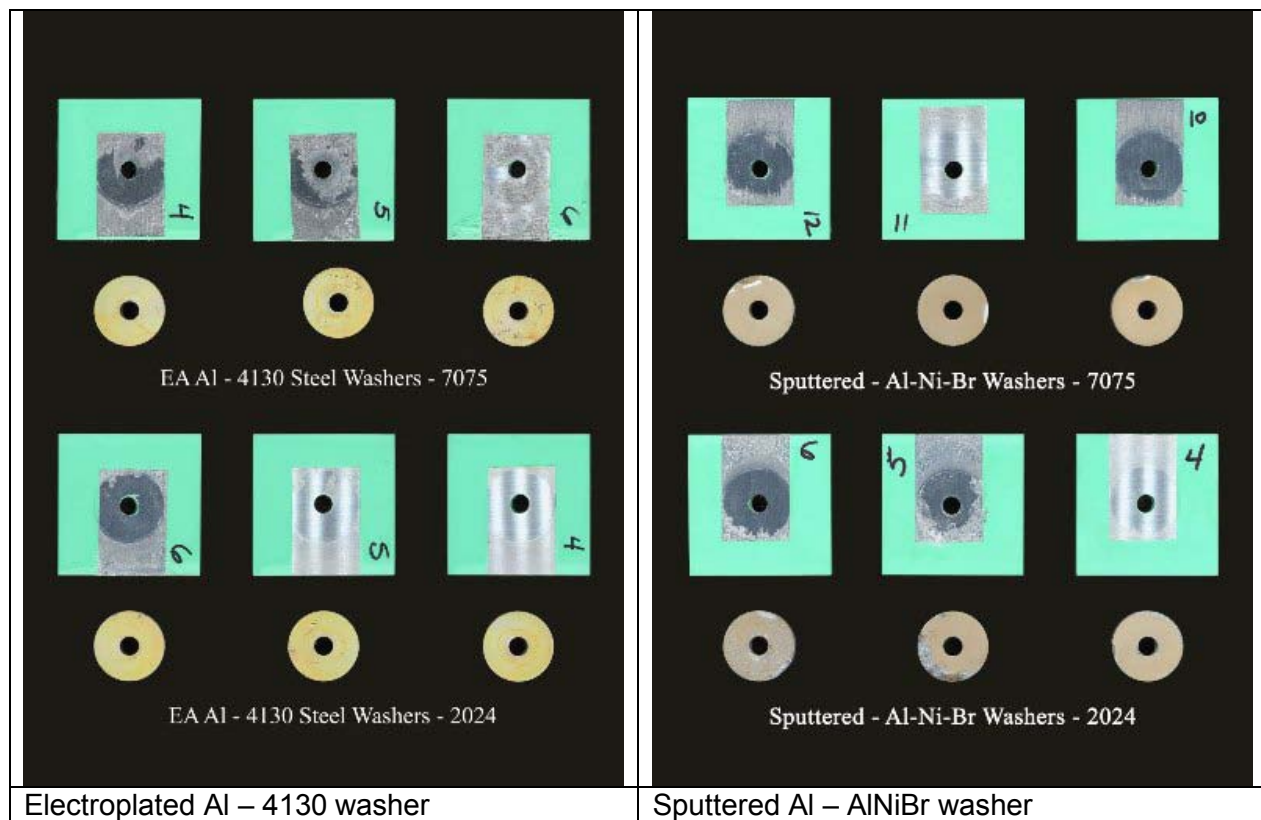


Figure 27. Representative Photos of ASTM G 85.A5 Cyclic Corrosion-Exposed Fixtures

6.1.3.3.4 Fluid Corrosion Resistance (HSS JTP 3.3.4)

Three 1 inch x 2 inches x 0.032 inch 4130 steel test specimens were cut and identified for each test fluid. Three test specimens were coated with each alternative coating, including any proposed conversion coatings to be used, to a minimum thickness of 0.3 mils. In addition, three test specimens were coated with Cd, meeting the requirements of MIL-STD-870B. The test specimens were cleaned and dried and then stored for a minimum of 16 hours in a desiccator over a suitable desiccant. Following desiccation, each specimen was then weighed to the nearest milligram (0.001 grams). Figure 28 shows a representative panel for each coating prior to immersion.

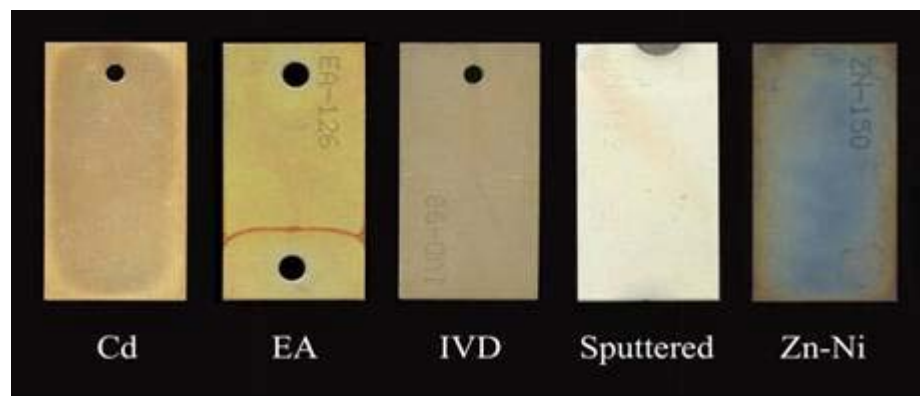


Figure 28. Representative Photos of Coated Panels Prior to Test

The specimens were tested IAW ASTM F 483 (Total Immersion Corrosion Test for Aircraft Maintenance Chemicals), except that the specimens were immersed for seven days. After immersion, the specimens were removed from the test fluid, cleaned with a suitable solvent, and dried in a desiccator for a minimum of 16 hours. After desiccation, the specimens were reweighed to the nearest milligram. The appearance of the specimens was then rated using the criteria described in ASTM F 1110 (Sandwich Corrosion Test), which is described in Table 57.

Table 57. ASTM F 1110 Corrosion Severity Rating System

Rating	Description
0	No visible corrosion (none)
1	Very slight corrosion or discoloration (up to 5% of the surface area corroded)
2	Slight corrosion (5 – 10%)
3	Moderate corrosion (10 – 25%)
4	Extensive corrosion or pitting (> 25%)

The following test fluids were utilized for the immersions:

- Reagent water (ASTM D 1193)
- Three parts by volume propylene glycol: one part distilled water
- Synthetic sea water (ASTM D 1141)
- Aircraft Deicing/Anti-Icing Fluid (SAE AMS 1424 / 1435)
- Runway deicing fluid (SAE AMS 1435)
- Cleaning compound, parts washer (MIL-C-29602)
- Cleaning compound, aerospace equipment (MIL-PRF-87937 Type I, Type II)
- Paint remover (MIL-R-81294)
- Paint remover (TT-R-2918 Type I [Turco 6813E])
- Paint remover (MIL-PRF-87978 Type I or equivalent)
- Paint remover – peroxide based
- Wheel well cleaning compound (MIL-PRF-85570 Type V)
- Water saturated MIL-PRF-8757 lubricant
- Water saturated MIL-PRF-5606 lubricant.

The following tables and figures list the average weight loss and appearance rating for each coating system exposed to each fluid. Appearance ratings for each coating are based upon comparison to the control specimens for that coating (see Figure 28). Figures 29 to 33 contain photos of the Cd-plated panels and each alternative after exposure to the test fluids. Table 58 contains the weight loss and appearance results. Table 58 quantifies the mass loss values and appearance results for each alternative coating in each test fluid, and furthermore contains two rows at the bottom with average mass loss values for each cadmium alternative (across all fluids), and for additional reference, aggregate totals of average mass loss added up for all fluids.



Figure 29. Cadmium Plated Panels after Fluid Immersion

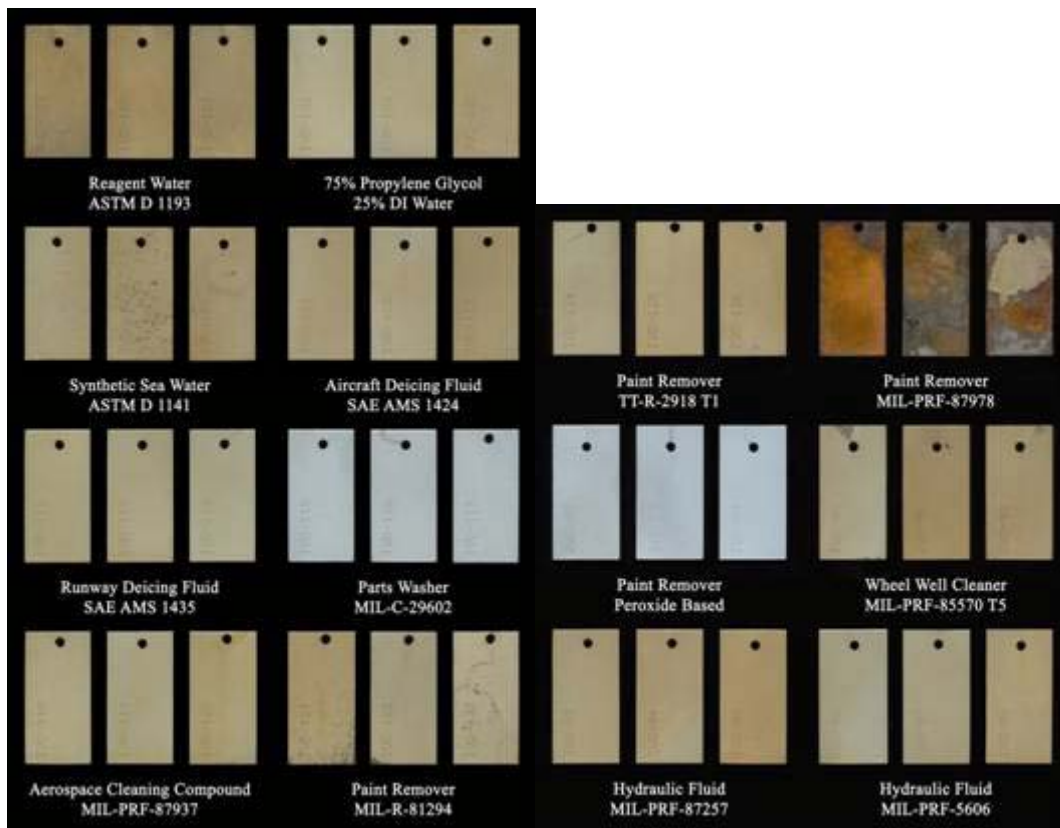


Figure 30. IVD-Aluminum Panels after Fluid Immersion



Figure 31. LHE Zn-Ni Panels after Fluid Immersion

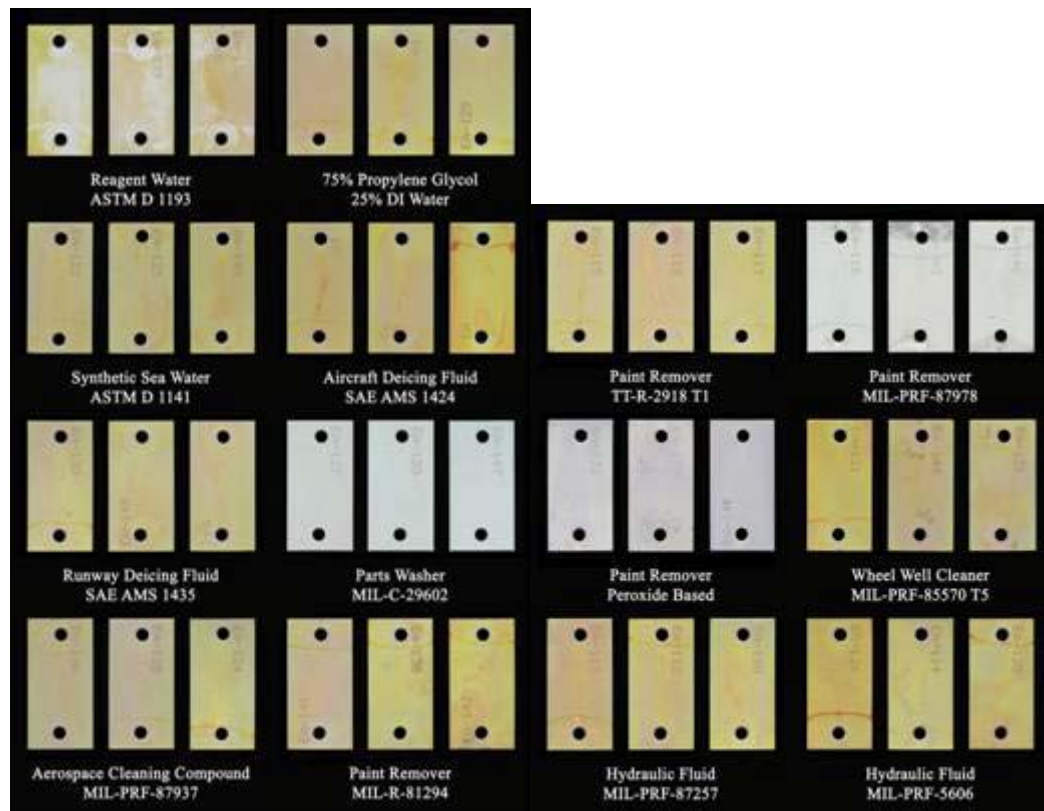


Figure 32. Electroplated Al Panels after Fluid Immersion



Figure 33. Sputtered Al Panels after Fluid Immersion

Table 58. Fluid Corrosion Resistance Test Results for Cd-plated Panels

Test Fluid	Cd-plated		IVD-Al		LHE Zn-Ni		Electroplated Al		Sputtered Al	
	Avg. Δ Mass	Appear. Rating	Avg. Δ Mass	Appear. Rating	Avg. Δ Mass	Appear. Rating	Avg. Δ Mass	Appear. Rating	Avg. Δ Mass	Appear. Rating
Reagent water (ASTM D 1193)	0.0015 grams (g)	2	0.0014 g	0	0.0001 g	0	0.0001 g	1	0.0097 g	0
3:1 Propylene glycol to water	0.0008 g	0	0.0006 g	0	0.0000 g	1	0.0003 g	0	0.0008 g	1
Synthetic sea water (ASTM D 1141)	0.0033 g	1	0.0016 g	2	0.0001 g	0	0.0002 g	0	0.0008 g	1
Aircraft Deicing/Anti-icing Fluid (SAE AMS 1425/1435)	0.0025 g	0	0.0001 g	0	0.0002 g	0	0.0003 g	0	0.1366 g	0
Runway Deicing Fluid (SAE AMS 1435)	0.0003 g	1	0.0004 g	0	0.0003 g	1	0.0002 g	0	0.0022 g	0
Cleaning Cpd, Parts Washer (MIL-C-29602)	0.0010 g	2	0.0013 g	2	0.0003 g	1	0.0001 g	0	0.0007 g	1
Cleaning Compound, Aerospace Equipment (MIL-PRF-87937 TyI/TyII)	0.0042 g	2	0.0005 g	0	0.0449 g	0	0.0001 g	0	0.0014 g	0
Paint Remover (MIL-R-81294)	0.0234 g	1	0.0004 g	1	0.0032 g	0	0.0003 g	0	0.2724 g	4
Paint Remover (TT-R-2918 Type I)	0.0029 g	2	0.0005 g	0	0.0470 g	3	0.0001 g	0	0.0006 g	0
Paint Remover (MIL-PRF-87978 Type I)	0.5235 g	4	0.3125 g	4	0.5920 g	4	0.0010 g	2	0.0000 g	2
Paint Remover – peroxide based	0.1869 g	4	0.0007 g	2	0.0570 g	4	0.0009 g	2	0.0007 g	2
Wheel well cleaning cmpd. (MIL-PRF-85570 Type V)	0.0015 g	1	0.0002 g	1	0.0415 g	0	0.0004 g	0	0.0011 g	1
Water Saturated MIL-PRF-87257	0.0010 g	0	0.0002 g	0	0.0001 g	0	0.0004 g	0	0.0155 g	0
Water Saturated MIL-PRF-5606	0.0004 g	0	0.0004 g	0	0.0001 g	1	0.0001 g	0	0.0003 g	0
AVERAGE REMOVAL, across all fluids (g)	0.0538		0.0229		0.0562		0.0003		0.0316	
Aggregate Total mass loss (g)	0.8070		0.3437		0.8430		0.0048		0.4738	

Of the alternative coating systems, the electroplated Al was by far the least affected by the fluids in terms of both appearance and mass loss. The bottom two rows in Table 58 show an approximate two order of magnitude improvement for electroplated Al when compared to each other alternative. For the baseline Cd-plated specimens and all other alternatives (excepted electroplated Al), the group of fluids that had caused the largest change in mass was the paint removers, clearly causing the most corrosion/removal of the coatings. The parts washer cleaning compound and the reagent water both appeared to remove the conversion coat on cadmium. The LHE Zn-Ni coating performed comparably to cadmium, with similar average mass removal values. The paint removers also had the most effect on weight and appearance. LHE Zn-Ni coating mass was also affected by the aerospace equipment cleaning compound and the wheel well cleaning compound.

Of the three high purity aluminum based coating systems, as stated earlier the electroplated Al was by far the least affected by the fluids, performing better than the IVD-Al baseline. Slight changes in weight were measured on the AlumiPlate™ for the MIL-PRF-87978 paint remover and peroxide-based paint remover, with visual examinations also showing some effect along with potential removal of the conversion coat. The parts washer cleaning compound also appeared to remove the conversion coat from the electroplated Al. The sputtered Al coating did not perform overall as well as IVD-Al. The MIL-R-81294 paint remover caused the sputtered Al coating to flake off of the panels, and changes in weight resulted from immersion in water-saturated MIL-PRF-87257 lubricant, reagent water, and deicing fluid.

6.1.3.3.5 Corrosion Resistance of Scribed Painted Coatings (HSS JTP 3.3.5)

The original JTP required only ASTM B 117 neutral salt fog testing for this performance parameter, but per later discussion between U.S. Army ARL and NAVAIR stakeholders a decision was made to include the accelerated corrosion test specification GM9540P for testing approximately half of the available panels since the Army heavily relies on this particular salt fog test. The GM9540P test includes additional salts as compared to ASTM B 117 and cycles through different corrosive conditions. Table 59 contains a succinct summary of corrosion resistance ratings and observations for the painted and scribed panels after 3000 hours of ASTM B 117 testing, while Table 60 contains the actual detailed evaluation of each test panel at each rating cycle. A few representative photos are shown in Figure 34. As listed in the “Panel #” column in Table 59, the following primers were tested, each using the same MIL-PRF-85285 polyurethane topcoat. Primers 1 and 2 contain chromate corrosion inhibitors. Primer 3 contained non-chromate inhibitors.

- Primer 1 (P1) = Deft MIL-PRF-23377, Class C2
- Primer 2 (P2) = Deft MIL-PRF-85582, Class C1
- Primer 3 (P3) = Deft MIL-PRF-85582, Class N

The relative corrosion resistance results for the painted panels were fairly similar to the results for the unpainted panels. The Cd-plated panels performed the best at the end of 3000 hours (Fig. 34, top left) although scribe rust had formed early on in the test for several panels (i.e. 9S ratings at 336 hours). LHE Zn-Ni was second best with three of five panels having a “9” rating after 3000 hours exposure (Fig. 34, top right). The other 2 panels were rated at 4 and 5 after 3000

hours due to pinhole rust and some creepage from the scribe, and there were some field blisters in the paint on the one painted with MIL-PRF-85582 Class C1. The electroplated Al panels were the next to follow in rank of performance. These panels were all exposed for 3000 hours, with final ratings of 0, 1, 3, and 5, due to various levels of creepage of corrosion from the scribe.

The sputtered Al panels were all pulled from testing after 1500 hours due to excessive rust formation; it was noted that the first signs of corrosion developed after 168 hours as creepage from the scribe. Finally, the IVD-Al baseline had four specimens with “0” ratings that were pulled from testing between 1000 to 2500 hours. One IVD-Al specimen, the only IVD-Al panel coated with solvent borne MIL-PRF-23377 primer, remained exposed for 3000 hours and received the highest rating of the group (“4”) rating based on the length of creepage from the scribe. Across all samples the solvent borne primer tended to provide the best corrosion protection.

**Table 59. Corrosion Resistance Results – Painted and Scribed Panels
(ASTM B 117, up to 3000 hours)**

Coating	Panel #	First Appearance of Corrosion/Observation	Time of Termination/Observation	Rating at Test Termination
Cd-plated	1 – P3	336 hours/ scribe rust	3000 hours/ scribe rust	9S
	2 – P3	336 hours/ scribe rust	3000 hours/ scribe rust	9S
	3 – P3	336 hours/ scribe rust	3000 hours/ scribe rust	9S
	4 – P2	168 hours/ scribe rust	3000 hours/ scribe rust	9S
	5 – P2	2000 hours/ scribe rust	3000 hours/ scribe rust	8S
IVD- Al(baseline – Hill AFB) IVD- Al(baseline – Hill AFB)	1 – P1	336 hours/some creepage from scribe	3000 hours/ 5 – 7 mm creepage from scribe	4
	2 – P2	168 hours/pinhole rust	2000 hours/ Excessive rusting	0
	3 – P2	168 hours/5 – 7 mm creepage from scribe	2500 hours/excessive pinhole rusting	0
	4 – P3	168 hours/pinhole rust with 3 – 5 mm creepage from scribe	1000 hours/excessive pinhole rusting	0
	5 – P3	168 hours/some creepage from scribe	2500hours/excessive scribe rust	0
LHE Zinc- Nickel	1 – P1	No rust	3000 hours/ Sacrificial coating breakdown/ no rust	9
	2 – P1	No rust	3000 hours/ Sacrificial coating breakdown/ no rust	9
	3 – P2	336 hours/pinhole rust	3000 hours/pinhole rust, 5 – 7 mm creepage	4P
	4 – P2	2500 hours/field blisters	3000 hours/field blisters	9FB
	5 – P3	2500 hours/pinhole rust with 2-3 mm creepage	3000 hours/pinhole rust with 3-5 mm creepage	5P
Electro- plated Al	1 – P2	168 hours/slight creepage	3000 hours/scribe rust with field blisters	0SFB
	2 – P2	168 hours/2-3 mm creepage	3000 hours/7-10 mm creepage	3
	3 – P2	168 hours/slight creepage	3000 hours/pinhole rust with 13-16 mm creepage	1P
	4 – P3	168 hours/slight creepage	3000 hours/7-10 mm creepage	3
	5 – P1	336 hours/2-3 mm creepage	3000 hours/3-5 mm creepage	5
Sputtered Al	1 – P3	336 hours/2-3 mm creepage	1500 hours/ Excessive rust	0
	2 – P2	168 hours/slight creepage	1500 hours/ Excessive rust	0
	3 – P1	168 hours/5-7 mm creepage	1500 hours/ Excessive rust	0
	4 – P2	168 hours/2-3 mm creepage	1500 hours/ Excessive rust	0
	5 – P3	168 hours/slight creepage	1500 hours/ Excessive rust	0

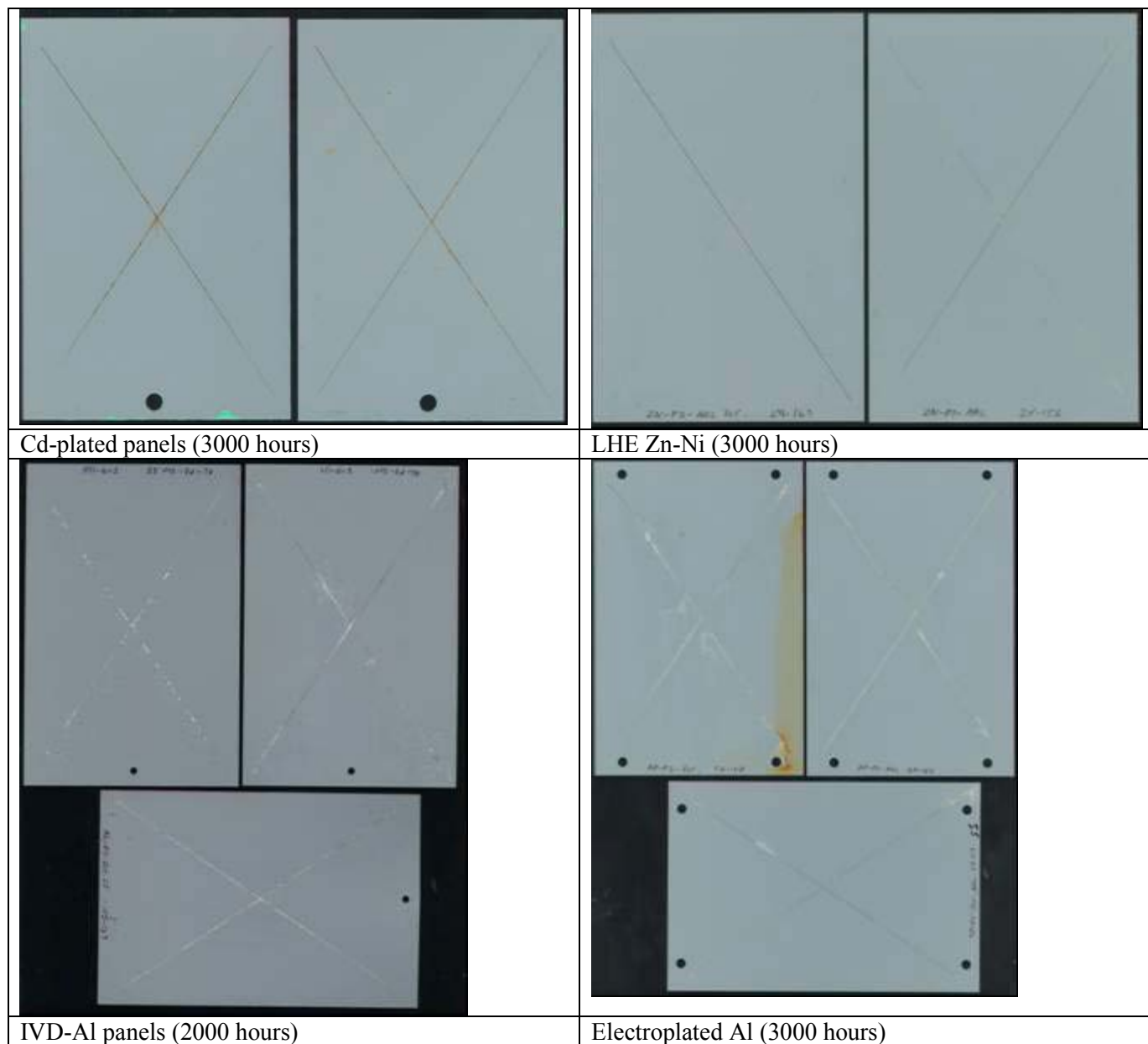


Figure 34. Representative Painted and Scribed Corrosion Resistance Panels

		
Sputtered Al panels (1500 hours)		

Figure 34 (cont'd). Representative Painted and Scribed Corrosion Resistance Panels

As referenced earlier, Table 60 contains the more detailed ASTM B 117 performance rankings and observations for each cadmium alternative plated panel with each of the three different primers used. Table 61 contains the analogous information for the panels tested in the GM9540P corrosion test preferred by the Army. These tables more graphically illustrate the progressive degradation in performance for each coating combination by sacrificial plating type as well as by primer type, which assist in quantifying trends. As with the ASTM B 117 testing, in GM9540 the Cd and LHE Zn-Ni coatings perform best, followed by the AlumiPlate™ coating, then IVD-Al and sputtered Al coatings. It is evident that primer P1 (MIL-PRF-23377 Class C2) performs best across the Cd alternative coatings in both types of accelerating salt fog testing.

Table 60. ASTM B 117 Corrosion Resistance for Painted and Scribed Cadmium Alternatives

Painted and Scribed		ASTM B 117 (Hours)							
Plating Type	Panel #, Primer	168	336	500	1000	1500	2000	2500	3000
Sputtered Al	1 - P3	10	6	3	1	0	-	-	-
Sputtered Al	2 - P2	7	4	3	1	0	-	-	-
Sputtered Al	3 - P1	4	4	4	2	0	-	-	-
Sputtered Al	4 - P2	6	4	3	1	0	-	-	-
Sputtered Al	5 - P3	7	5	4	3	0	-	-	-
IVD-Al	1 - P1	9	7	7	7	7	6	5	4
IVD-Al	2 - P2	6P	5	4	3	1	0	-	-
IVD-Al	3 - P2	4	3P	3P	3P	3P	2P	0P	-
IVD-Al	4 - P3	5P	3P	2P	0P	-	-	-	-
IVD-Al	5 - P3	6	6	5	5	3S	1S	0S	-
AlumiPlate	1 - P2	8	7	5SFB	4SFB	4SFB	3SFB	3SFB	0SFB
AlumiPlate	2 - P2	6	6	6	5	5	4	4	3
AlumiPlate	3 - P2	7	5	5P	5P	4P	3P	3P	1P
AlumiPlate	4 - P3	7	7	5	5	5	5	3	3
AlumiPlate	5 - P1	9	6	6	6	6	5	5	5
LHE Zn-Ni	1 - P1	10	10	10	10	9	9	9	9
LHE Zn-Ni	2 - P1	10	10	9	9	9	9	9	9
LHE Zn-Ni	3 - P2	9	9P	9P	9P	9P	9P	5P	4P
LHE Zn-Ni	4 - P2	9	9	9	9	9	9	9FB	9FB
LHE Zn-Ni	5 - P3	10	10	10	10	10	10	6P	5P
LHE Cadmium	1 - P3	9	9S	9S	9S	9S	9S	9S	9S
LHE Cadmium	2 - P3	10	9S	9S	9S	9S	9S	9S	9S
LHE Cadmium	3 - P3	10	9S	9S	9S	9S	9S	9S	9S
LHE Cadmium	4 - P2	9S	9S	9S	9S	9S	9S	9S	9S
LHE Cadmium	5 - P2	10	10	10	10	10	9S	9S	8S

Note: P = Pinhole rust; S = Scribe rust; FB = Field Blisters (blisters in paint away from scribe).

Table 61. GM9540P Corrosion Resistance for Painted and Scribed Cadmium Alternatives

Painted, Scribed Test Panels		GM9540P Cyclic Corrosion, Cycles								
Plating Type	Primer #	10	20	40	60	80	100	120	160	200
Sputtered Al	P2	9S	7S	7S	7S	5S	5S	4S	0S	
Sputtered Al	P3	8S	8S	8S	7S	6S	5S	3S	3S	3S
Sputtered Al	P2	7S	6S	4S	4S	2S	0S			
Sputtered Al	P3	7S	5S	5S	4S	3S	3S	3S	0S	
Sputtered Al	P1	8S	7S	7S	6S	6S	5S	4S	0S	
IVD Al	P3	8S	7S	6S	5S	4S	3S	3S	0S	
IVD Al	P1	10	9S	6S	5S	5S	3S	2S	0S	
IVD Al	P2	9S	5S	2S	0S					
IVD Al	P	8S	7S	6S	6S	5S	5S	5S	3S	2S
IVD Al	P3	8S	7S	6S	5S	4S	3S	3S	0S	
AlumiPlate	P1	10	10	10	9	9	9	9	9	9
AlumiPlate	P2	10	10	10	10P	10P	10P	10P	10P	10P
AlumiPlate	P2	10	10	10	9	9	6	6	5S	5S
AlumiPlate	P3	10	10	10	10	10	10	10	10	10
AlumiPlate	P3	10	10	10	10	10	9	9	9S	9S
LHE Zn-Ni	P2	10	10	10	10	10	10	10	10	10
LHE Zn-Ni	P2	10	10	10	10	10	10	10	10	10
LHE Zn-Ni	P1	10	10	10	10	10	9	9	9	9
LHE Zn-Ni	P2	10	10	10	10P	10P	10P	10P	10P	10P
Cadmium	P1	10	10	10	10	10	10	10	10	10P
Cadmium	P	10P	10P	10P	10P	10P	10P	10P	10P	10P
Cadmium	P2	10	10	10	9S	9S	9S	9S	9S	9S
Cadmium	P1	10	10	10	10	10	10	10	10	10
Cadmium	P	10	10	10	10	10	10	10	10	10

Note: P = Pinhole rust; S = Scribe rust.

6.1.3.4 Lubricity (HSS JTP 3.4)

Cadmium alternative coatings were measured for the necessary lubricity properties when applied to threaded fasteners. These properties included run-on and breakaway torque, and torque tension. Results are presented for these tests in 6.1.3.4.1 and 6.1.3.4.2, respectively.

6.1.3.4.1 Run-on and Breakaway Torque (HSS JTP 3.4.1)

The maximum locking torque was measured after two (2) complete turns (720° rotation) from the point where the top of the nut is flush with the end of the bolt. The maximum locking torque was the highest reading obtained during the third full turn (360° rotation). The breakaway torque

was then measured during removal of the nut from the clamped up threaded part. The nut was removed from the test bolt between each cycle. In addition, any loosened particles were blown off with compressed air, if necessary, before continuing. The locking and breakaway torque was measured for 15 lock/breakaway cycles and at completion of the testing, the nut and bolt were examined for thread damage at ten (10) times magnification.

Figures 35 through 38 contain graphs of the maximum locking torque and breakaway torque for each size fastener, with the alternative coating systems and Cd baseline. Note that the acceptance criterion for the 3/8-inch fastener is a locking torque of less than 80 in-lb and a minimum breakaway torque of 9.5 in-lb. The criterion for the 5/8-inch fastener is a locking torque of less than 300 in-lb and a breakaway torque of more than 32 in-lb.

Figures 35 and 36 represent the testing performed on the 3/8-inch fasteners. All coatings met both the maximum locking and breakaway torque acceptance criteria. The locking torque results stabilized for all coatings after approximately the fifth cycle with the LHE Zn-Ni having the lowest locking torque values and the sputtered Al coating had the highest. Also, the shapes of the curves and trends for locking torque closely matched the curves for breakaway torque, with the only difference being that the breakaway torque values were lower than the locking torque values, which was expected.

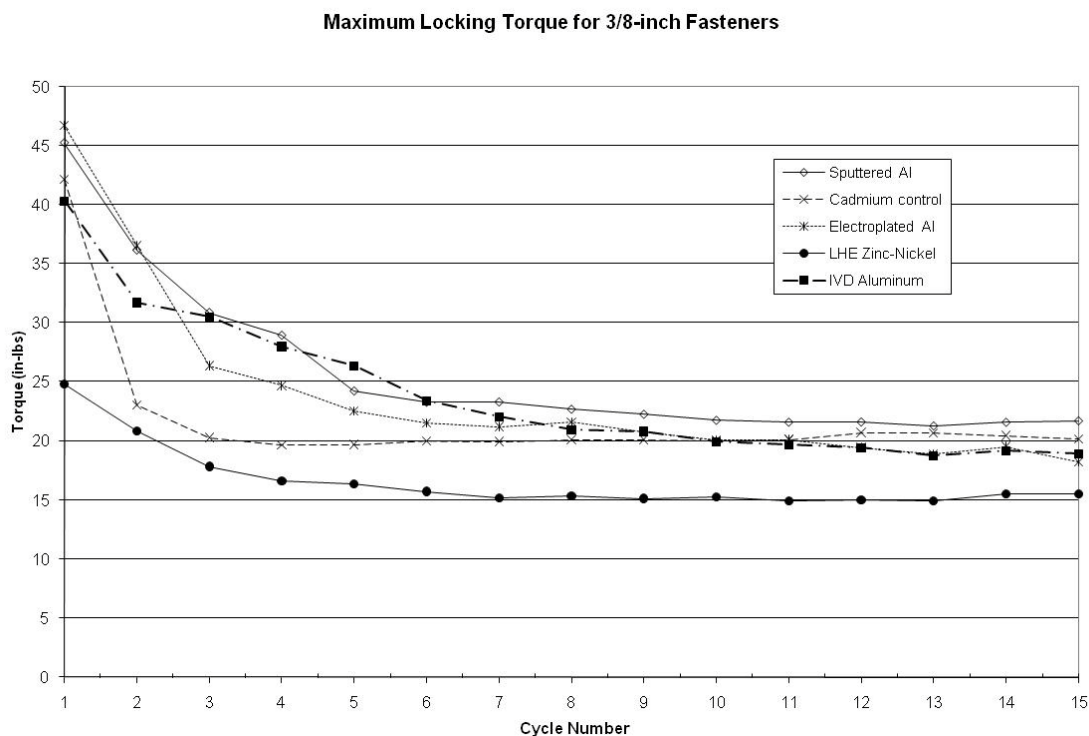


Figure 35. Graph of Maximum Locking Torque for the 3/8-inch Fasteners

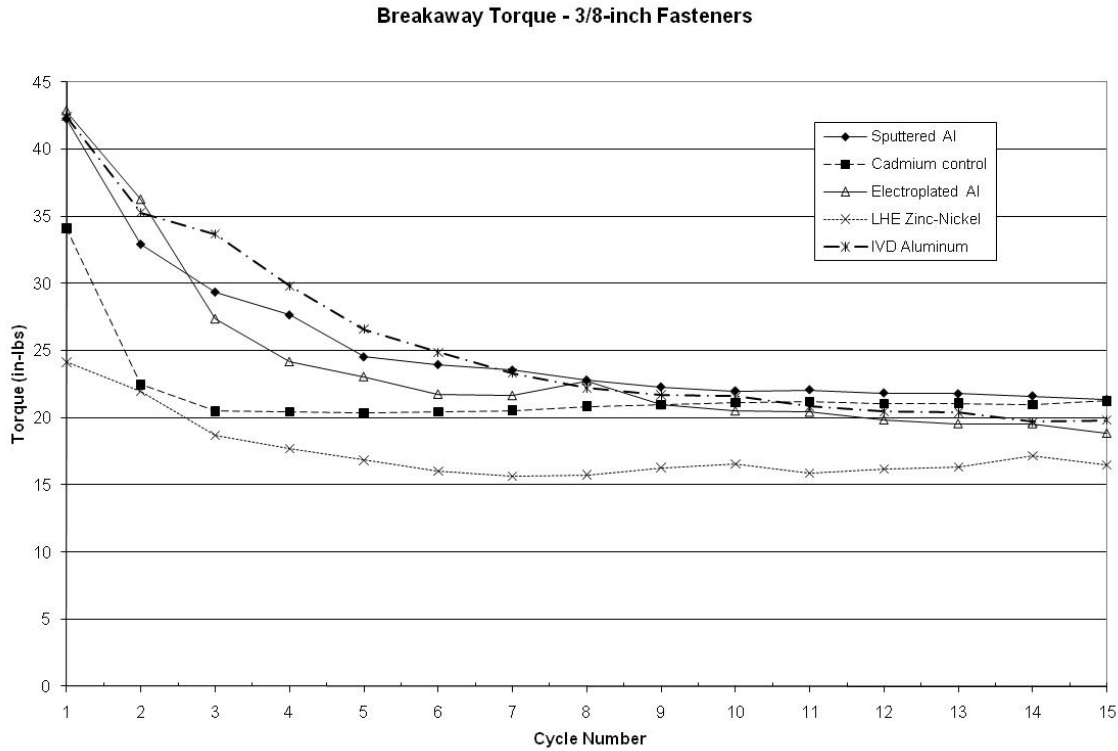


Figure 36. Graph of Breakaway Torque for 3/8-inch Fasteners

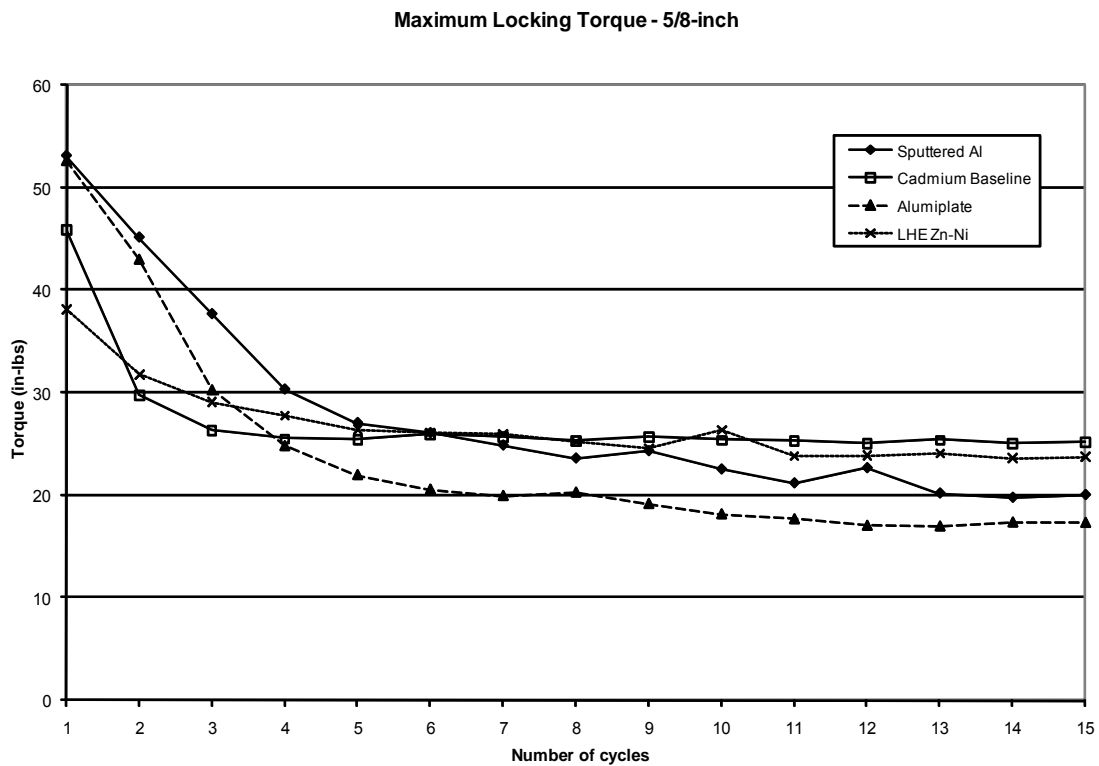


Figure 37. Graph of Maximum Locking Torque for 5/8-inch Fasteners

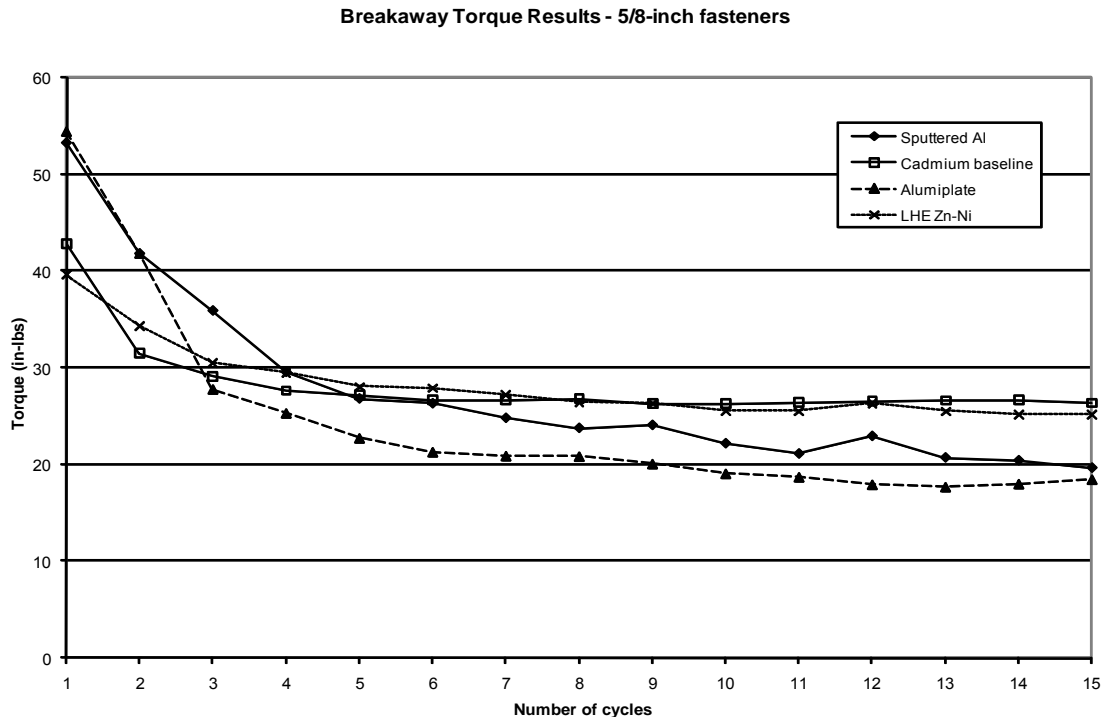


Figure 38. Graph of Breakaway Torque for 5/8-inch Fasteners

For the 5/8-inch fasteners, the Cd baseline and the LHE Zn-Ni had the highest maximum locking torque readings, once the readings stabilized at the sixth cycle (Fig. 37). All results met the acceptance criteria ≤ 300 in-lb). The breakaway torque readings, however, did not meet the acceptance criterion of being greater than 32 in-lb. All coatings were less than 32 in-lb of torque after the second cycle, with the Cd baseline and LHE Zn-Ni having consistent results between 25 and 30 in-lb of torque. These values would likely be higher with other thread treatments, as they probably represent minimum values due to the anti-seize lubricant specified for testing in Phase II of the JTP.

6.1.3.4.2 Torque Tension (HSS JTP 3.4.2)

This test measured the torque-tension values during installation of threaded fasteners; torque-tension is important to achieve the specified clamp-up force (load) within the assembly. Candidate coatings were applied as recommended by the manufacturer to the same thickness class as the LHE Cd baseline. A representative test fixture to measure torque-tension is shown in Figure 39.

The test nut, bolt, and washers were tested per the original JTP recommendation of using an anti-seize product IAW SAE AMS 2518 (Thread Compound, Anti-Seize, Graphite-Petrolatum, revised July 2001) using the method listed in Table 62. Graphite based lubricants are not recommended for use in corrosive environments; however, it provided a consistent basis to compare cadmium with its alternatives. It should be noted that multiple other thread treatments are used in DoD systems and they may exhibit different relative performance than shown here.

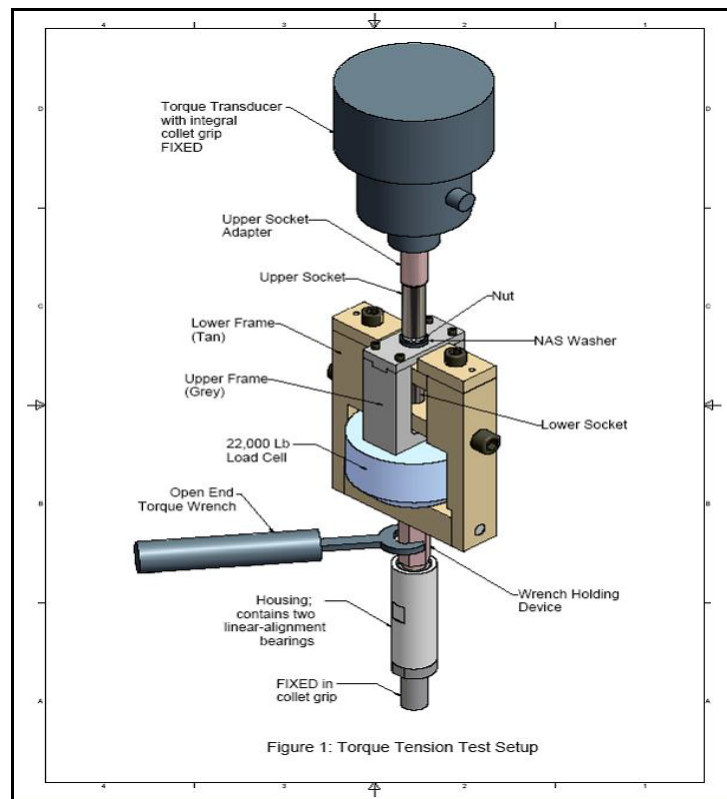


Figure 39. Torque-Tension Test Fixture

The nut was assembled onto the bolt so that a minimum of one complete thread extended beyond the top of the nut. The torque-tension was measured using the recommended torque transducer and force washer. The torque and induced load was recorded for the range of 30% to 60% of the ultimate tensile strength (UTS) of the bolt (3,435-6,870 lb for -06032 bolts, 10,239-20,478 lb for -10032 bolts).

Each assembly was tested for a total of five cycles. A test cycle included wrenching nut onto bolt until the desired preload was achieved, then completely removing nut. Any loosened particles were blown off using 60 psi compressed air, if necessary, between cycles. Representative graphs for each alternative coating are presented as Figures 40 through 43 for the torque tension, load versus torque curves for the 3/8-inch fasteners. Each graph contains data for 5 cycles of loading and then completely removing the nut. Visual observation provided by WMTR was that there was no loose/peeling coating or stripping of the threads on the bolts during testing.

Table 62. Test Method for Torque Tension

Parameters	Room temperature (68–78°F), installation torque range of 50–75 in-lb
Type/Number of Specimens	Test Bolts/Nuts: NASM21250-06032/ NAS1804-6 (alloy steel, candidate coated, 3/8 inch), five (5) specimens

	Test Bolts/Nuts: NASM21250-10032/ NAS1804-10 (alloy steel, candidate coated, 5/8 inch), five (5) specimens Test Washers: NASM14155-6 or NASM14155-10 (alloy steel, candidate coated) two (2) per test specimen
Experimental Control Specimens	Test Bolts/Nuts: NASM21250-06032/ NAS1804-6 (alloy steel, Cd coated, 3/8 inch), five (5) specimens Test Bolts/Nuts: NASM21250-10032/ NAS1804-10 (alloy steel, Cd coated, 5/8 inch), five (5) specimens Test Washers: NASM14155-6 or NASM14155-10 (alloy steel, Cd coated) two (2) per test specimen
Acceptance Criteria	Torque-tension for candidate material is within the range for LHE Cd. Threads do not yield, fracture, or strip.
Reference Document	MIL-STD-870B, AMSQP416

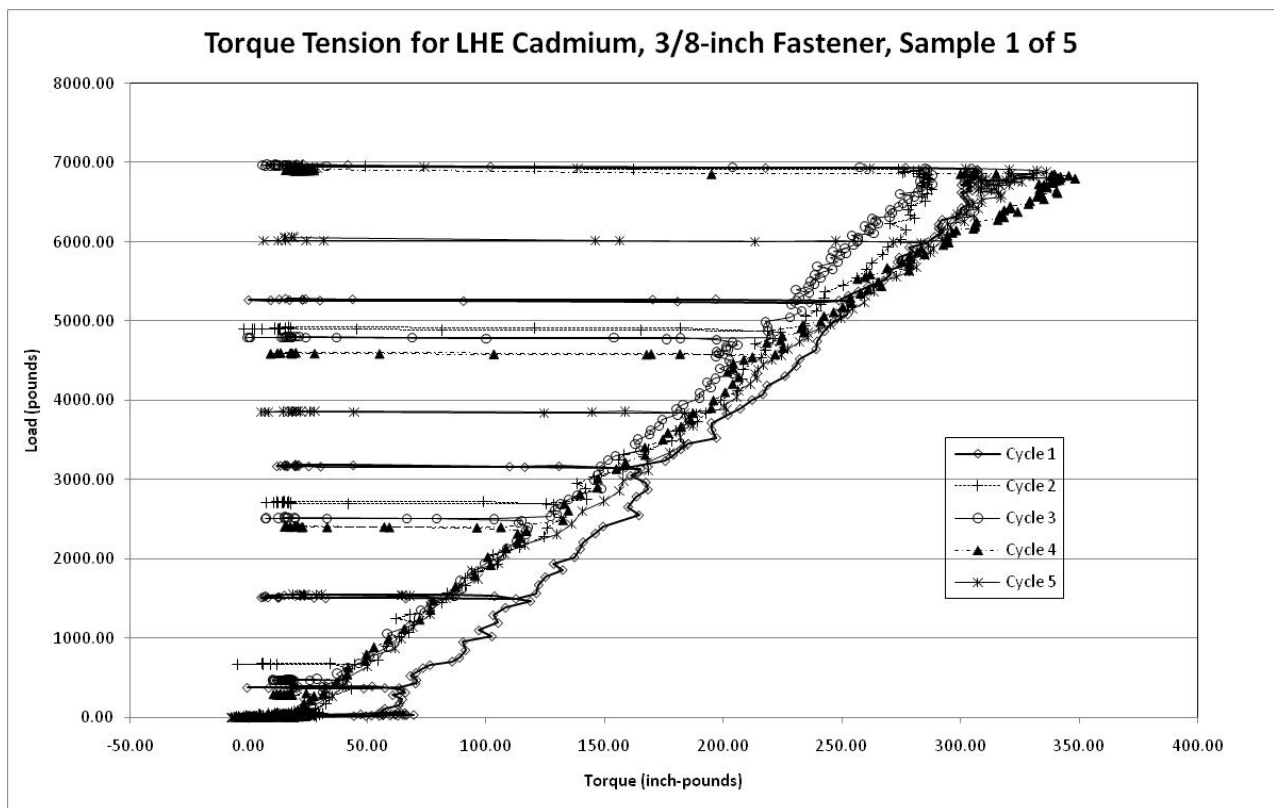


Figure 40. Torque Tension Results for LHE Cd, 3/8-inch Fastener

The load versus torque curves for each of the five cycles for LHE cadmium is provided in Figure 40. There is a dataset for each of five loading cycles for the same fastener and each is plotted with discrete symbols. The ideal results for this test entail linear load development as torque is applied to the system, and similar values of torque required for application of the target load for each cycle. For the LHE cadmium coating, Figure 40 illustrates that there are relatively linear

loading curves and that there is minimal change in the required torque values among the five cycles. This indicates that the coating remained intact and continued to provide relatively consistent lubricity and load transfer throughout the five cycle test. A ‘drop-in’ alternative to cadmium on fasteners would have either similar torque-tension curves, or to have torque-tension properties modified by a suitable thread treatment to place its performance within the typical range of cadmium.

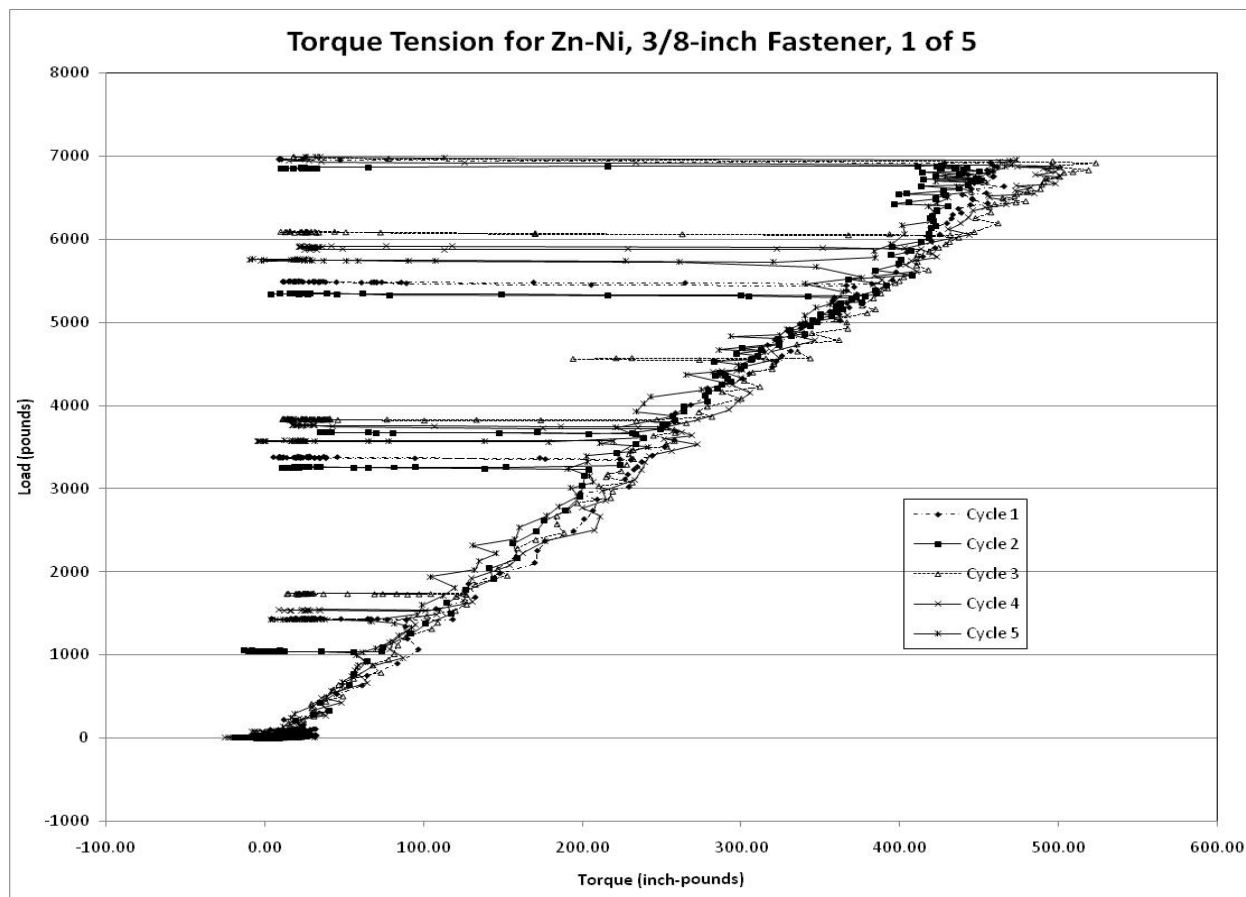


Figure 41. Torque Tension Results for Zn-Ni Coated 3/8-inch Fasteners

Figure 41 displays the results for the Zn-Ni coated 3/8-inch fasteners. Of the alternatives tested in this project, the LHE Zn-Ni torque-tension repeatability was most similar to LHE cadmium in that there is comparable variance in peak torque values from Cycle 1 to Cycle 5. The absolute values of the maximum torque were higher than cadmium by a fairly significant percentage, however, ranging from approximately 400—500 in-lbs as compared to 275—350 in-lbs for cadmium. This difference could result in preload differences in the ~35% range lower than comparable LHE cadmium plated hardware.

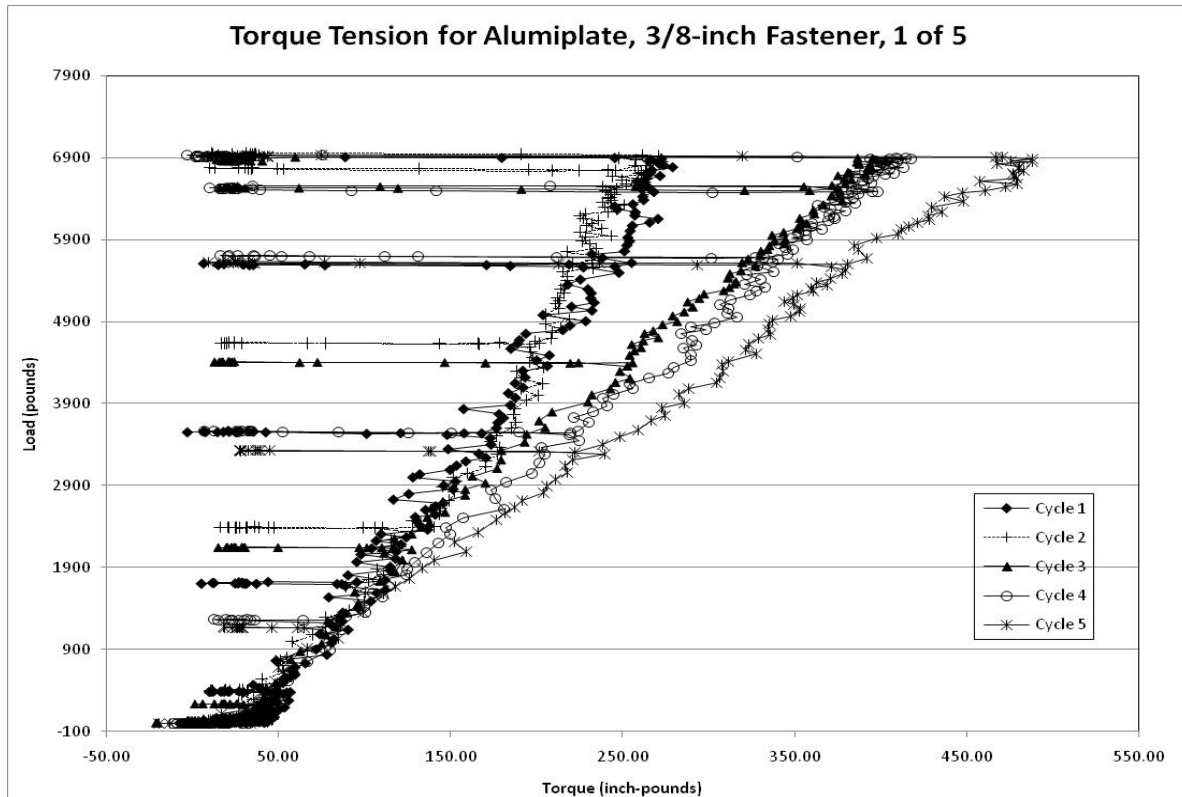


Figure 42. Torque Tension Results for 3/8-inch Fasteners Coated with Electroplated Al

Figure 42 contains the curves for electroplated Al on 3/8-inch fasteners. For the electroplated Al coated fasteners, the torque values increased after the second cycle becoming somewhat higher than the LHE Cd baseline torque values (Fig. 40), indicating that the electroplated Al decreased in lubricity through wear. Again, all curves are relatively linear during the loading cycle.

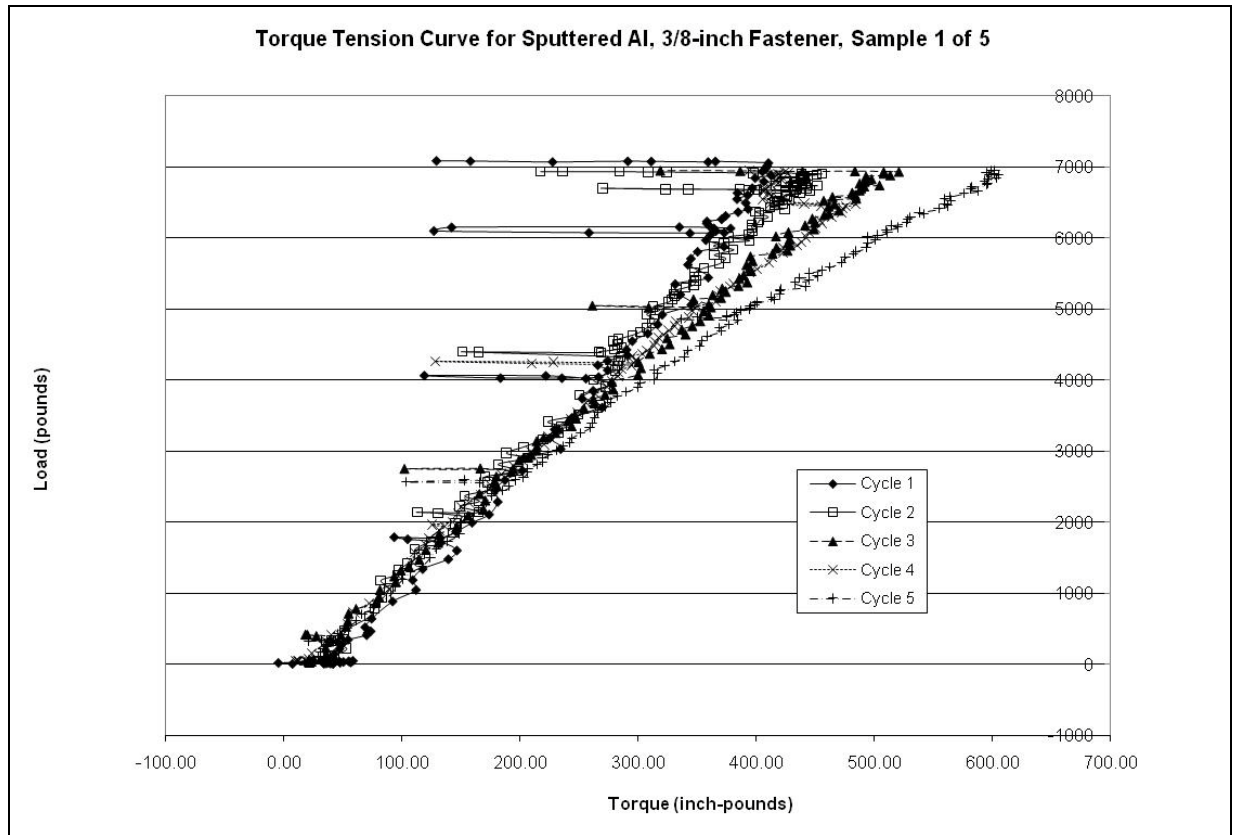


Figure 43(a). Torque Tension Results for 3/8-inch Fasteners Coated with Sputtered Al

Figure 43(a) shows the results for sputtered Al-coated 3/8-inch fasteners. The curves for sputtered Al show the same trend as the electroplated Al (Fig. 42), with torque values increasing during loading from Cycle 1 to Cycle 5. The maximum torque values required for sputtered Al (400–600 in-lbs) are slightly higher than electroplated Al (250–475 in-lbs).

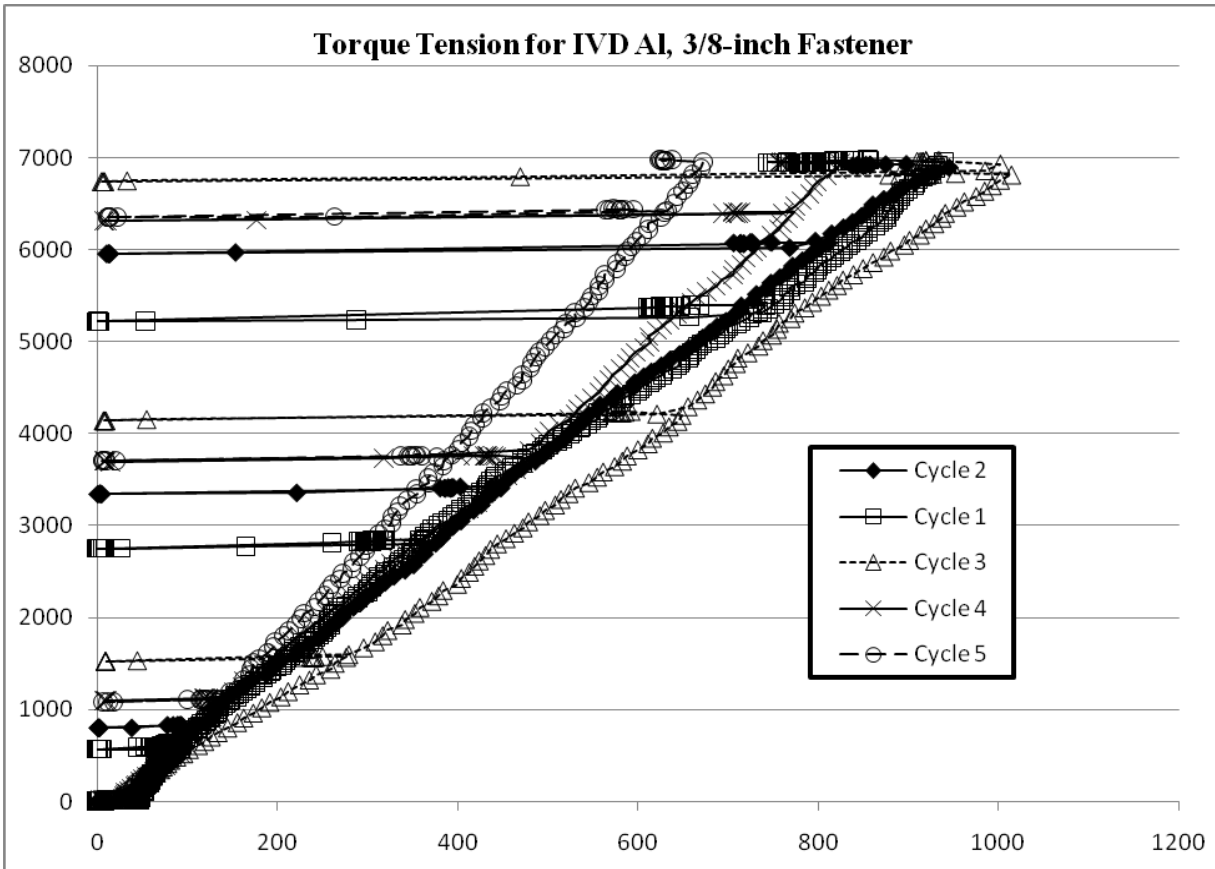


Figure 43(b). Torque Tension Results for 3/8-inch Fasteners Coated with IVD-Al
(Axes are the same as Fig. 43(a))

The curves for IVD-Al (Fig. 43(b)) indicate the coating has less lubricity than the other aluminum coatings as observed by the significantly higher required torque values, most of them in the 800-1000 in-lb range except for the fifth cycle which was lower. These values are higher than sputtered Al (400–600 in-lbs) and electroplated Al (250–475 in-lbs). There was no visible loss of the IVD-Al coating after the tests.

The next series of graphs (Figures 44–47) represents the results achieved for the cadmium alternative coatings on 5/8-inch fasteners.

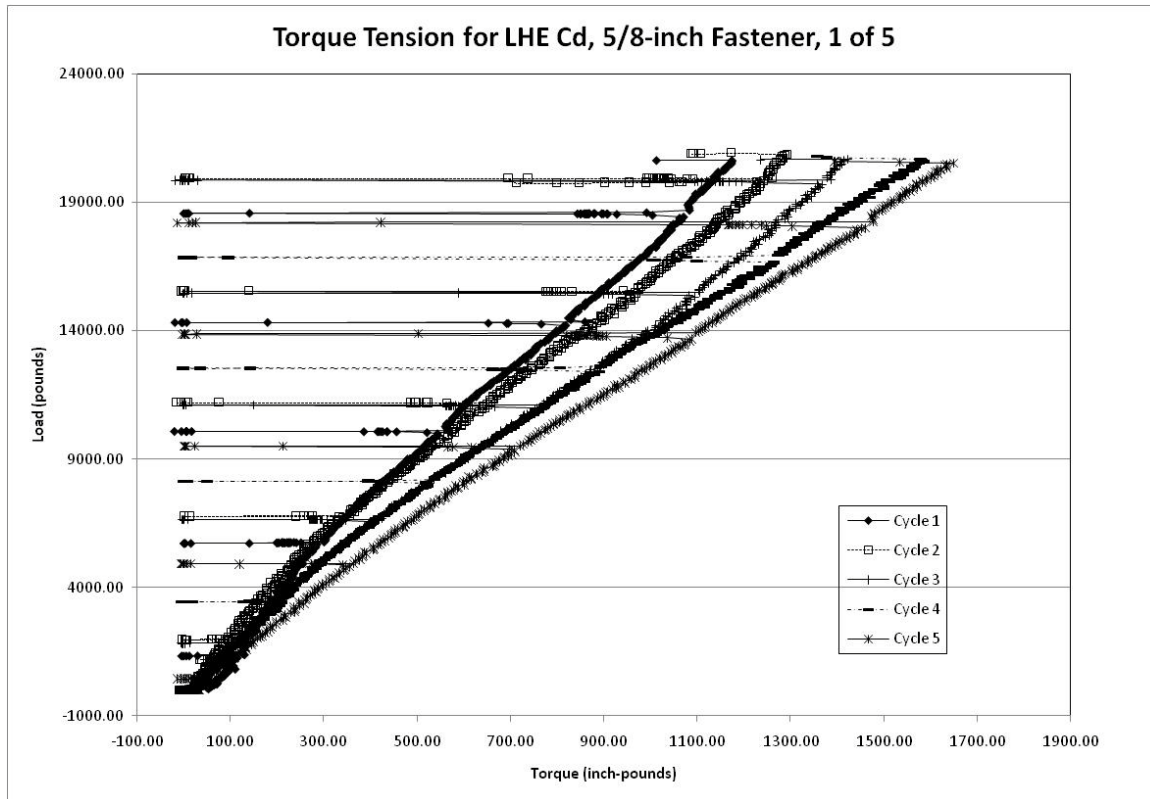


Figure 44. Torque Tension Results for LHE Cd-coated 5/8-inch Fastener

The results for LHE cadmium on a 5/8-inch fastener are presented in Figure 44. The results show a trend of increasing torque required to achieve the target load, indicating a gradual loss in lubricity. These results show somewhat more variability in torque values than the cadmium coated 3/8-inch fasteners (Fig. 40).

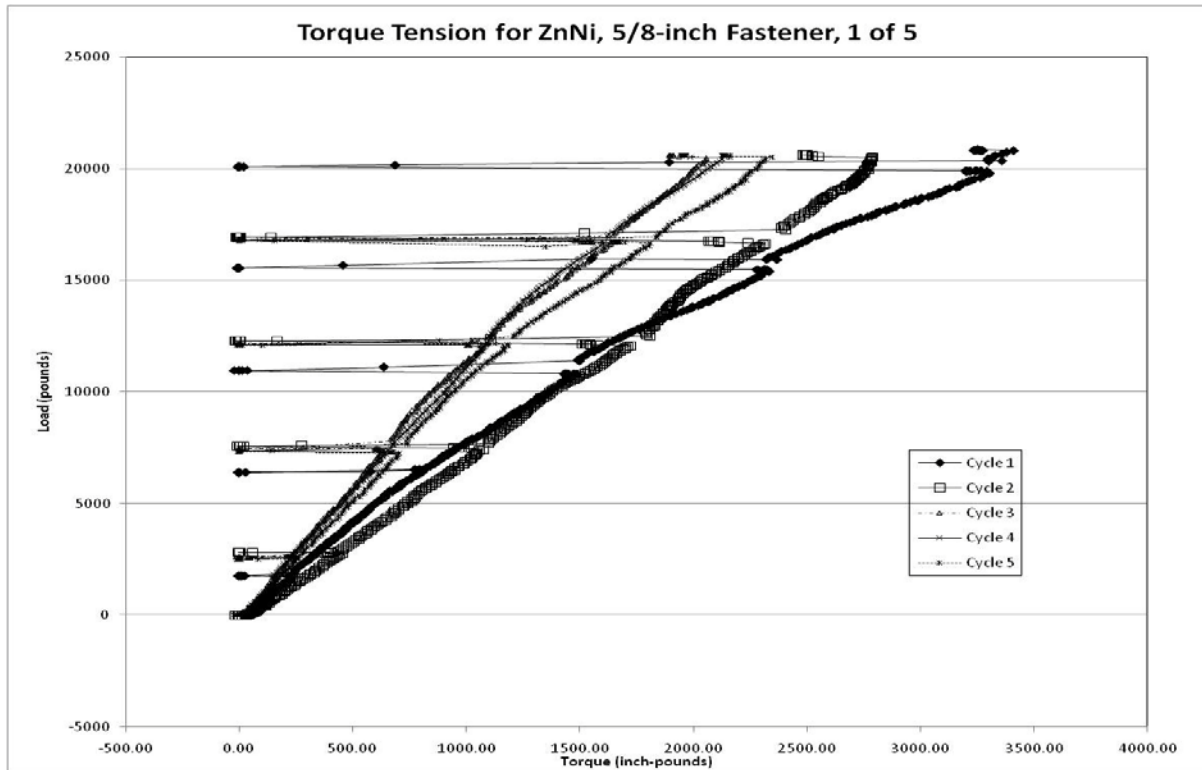


Figure 45. Torque Tension Results for 5/8-inch Fastener Coated with ZnNi

Figure 45 contains a representative graph for the Zn-Ni coated 5/8-inch fasteners. Required torque values were in the 2000-3300 in-lb range, substantially higher than LHE Cd (1200—1600 in-lb as shown in Fig. 44). The results for the 5/8-inch fasteners coated with LHE Zn-Ni also exhibited a trend of increased lubricity with each applied cycle, which is opposite LHE cadmium on the 5/8-inch fastener (Fig. 44).

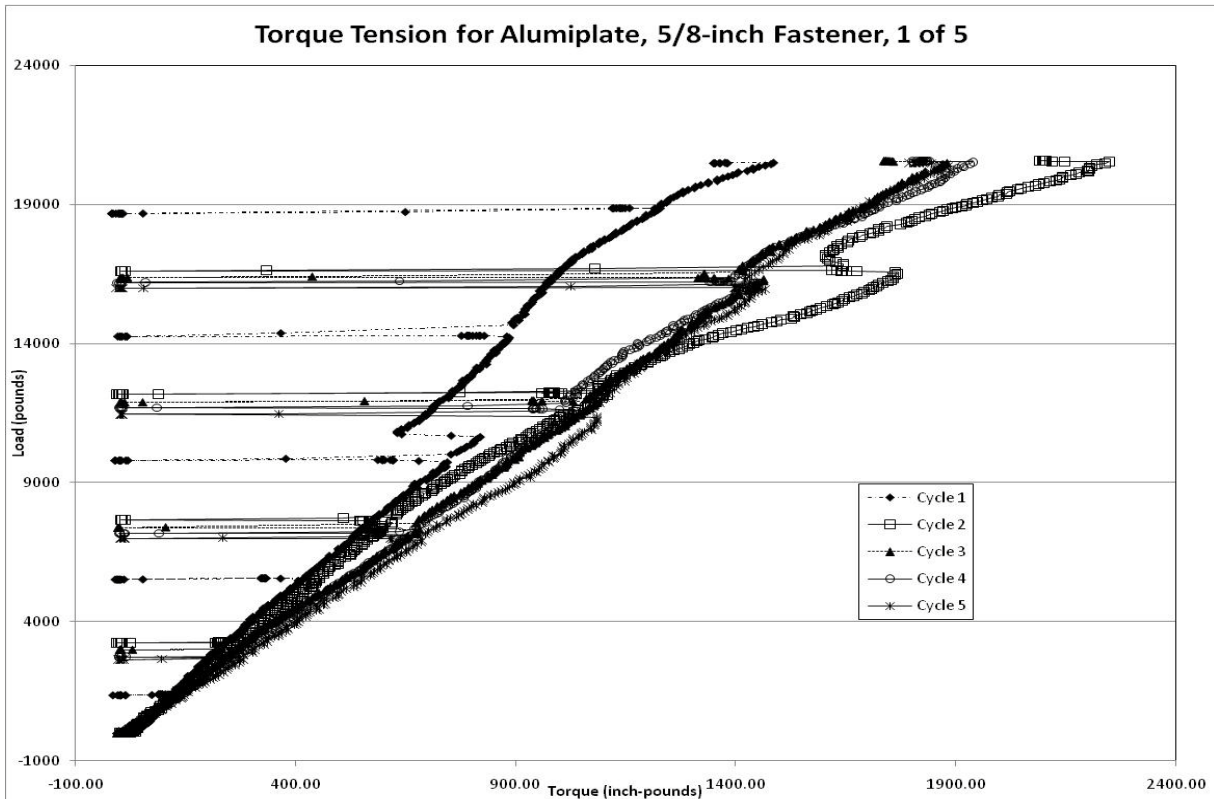


Figure 46. Torque Tension Results for 5/8-inch Fastener Coated with Electroplated Al

Figure 46 shows the load vs. torque plots for a 5/8-inch fastener coated with electroplated Al. This loading curves are less linear than the 3/8-inch fasteners, with some irregularities as shown. showing a lower load versus torque curve for Cycle 1, then the highest torque readings for Cycle 2, with the last three cycles having curves between the first two.

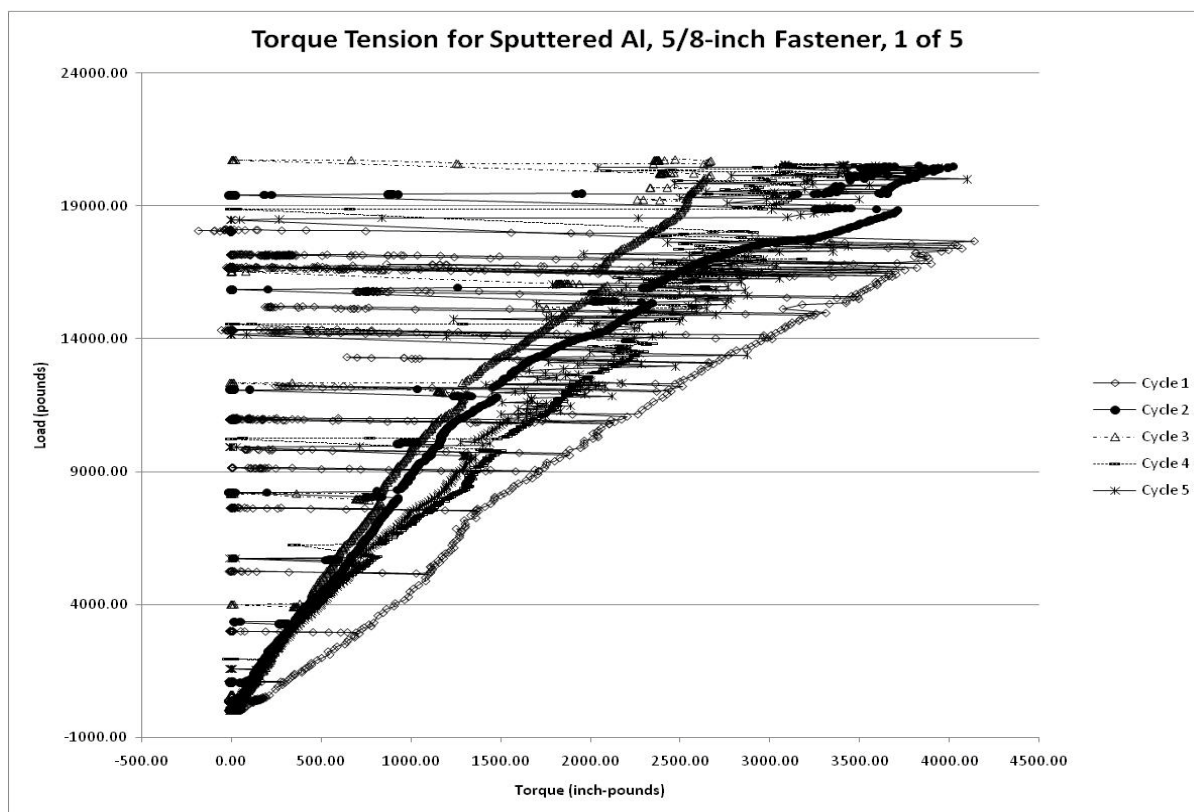


Figure 47(a). Torque Tension Results for 5/8-inch Fastener Coated with Sputtered Al

Figure 47(a) contains a graph of the sputtered Al torque tension results. The results for the 5/8-inch fasteners coated with sputtered Al show many more drop points on the curves than the previous samples. Also, the trend is not clearly defined, similar to the electroplated Al results (Fig. 46). For the particular fastener represented, there appeared to be a great deal of scatter to the Cycle 4 and Cycle 5 curves at torque values exceeded 1500 – 2000 in-lbs. There does appear to be more of an effect on the coating when conducting torque tension testing on the 5/8-inch fasteners as opposed to the 3/8-inch fasteners.

The results for the 5/8-inch fasteners coated with IVD-Al showed that torque values in the 4000-4500 in-lb range were required to achieve the target load (Fig. 47(b)). The IVD-Al fasteners showed more consistency than the other aluminum coatings on the 5/8-inch fasteners, but required substantially higher torque values.

Overall, the torque tension results showed that the LHE cadmium and LHE Zn-Ni coatings generally exhibited the most consistent torque-tension values over the 5 cycles of installation and removal which is best for maintaining consistency in target loads on a particular joint. The aluminum coatings tended to exhibit more variability in required torque to achieve the desired load when using torque values as a guide. The aluminum coatings also displayed less linearity in loading especially on the larger (5/8-inch) fastener size in these experiments.

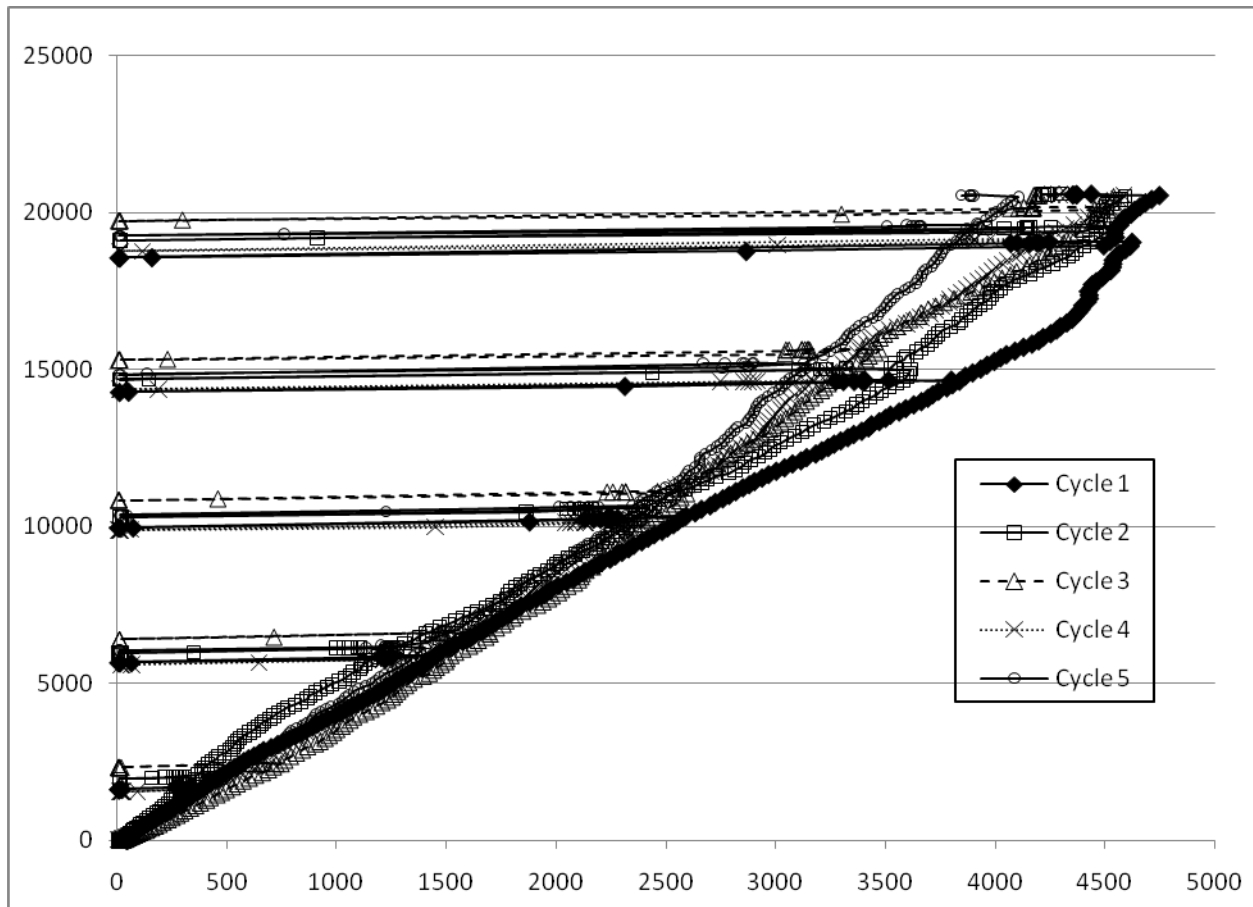


Figure 47(b). Torque Tension Results for 5/8-inch Fastener Coated with IVD-Al
(Axes are the same as Fig. 47(a))

6.1.3.4.3 Torque Tension for Corrosion Exposed Fasteners (HSS JTP 3.4.3)

At the beginning of this effort a test facility could not be located to perform this testing (a number of no-bid responses were received) therefore it was not completed within this project. The effect of corrosion on the torque-tension response is still of interest for any potential cadmium replacement coating to determine the relative changes that occur as corrosion proceeds, including potential to lock or seize.

6.1.3.5 Fatigue (HSS JTP 3.5)

6.1.3.5.1 Rotating Beam Fatigue (HSS JTP 3.5.1)

Rotating Beam Fatigue tests were to be conducted in Phase III of this ESTCP effort per the original JTP execution plan, however, Phase III was not performed. See Sec. 6.1.4.1.4 for the results of axial fatigue tests performed in the Navy Added portion of the JTP.

6.1.3.6 Hydrogen Embrittlement and Re-Embrittlement (HSS JTP 3.6)

6.1.3.6.1 Hydrogen Embrittlement (HSS JTP 3.6.1)

Hydrogen embrittlement testing was included in both test phases as a quality assurance measure. In this test ASTM F 519 Type 1a.1, 4340 high strength steel notched round bars were loaded in tension for an extended period of time to determine whether the coating process was embrittling. Each coated bar was loaded for 200 hours at 75% of the notch fracture strength (NFS) established for uncoated bars of the same lot. The tensile load was subsequently increased by 5% per hour (in a stepwise fashion) until bar fracture. Four replicates of each coating were tested. The acceptance criteria for this test were the NFS of bare and coated bars be within 10 ksi of the average reported by the manufacturer for bare bars, and four of four bars sustain 75% NFS for 200 hours without fracture; OR only one of four bars fracture in less than 200 hours and the remaining three sustain at least one hour at 90%. The constant rate tensile pull test, used to determine whether the coated test bars fracture within 10 ksi of bare bars as reported by the manufacturer, has generally not been conducted industry-wide in the past and was not conducted in this effort except as described below. A photograph of the test equipment is shown in Figure 48 and test results are shown in Table 63 for both primary and repair coatings (Phase I only).



Figure 48. Hydrogen Embrittlement Test Equipment

All coatings passed this Phase I test except Sn-Zn and brush Zn-Ni. The failure mode was evaluated for two of seven Sn-Zn bars. Both Sn-Zn bars had single large intergranular fracture areas initiated at the notch surface, indicating the Sn-Zn plating process embrittled the steel. The failure was discussed with the coater who recommended a nickel strike to mitigate embrittlement. The JCAT previously established that a nickel strike was not permitted for this test effort, so this was not pursued for Sn-Zn. Passing hydrogen embrittlement testing was a prerequisite for hydrogen re-embrittlement testing. Since Sn-Zn did not pass hydrogen embrittlement testing the coating was dropped from further evaluation.

Table 63. Hydrogen Embrittlement Test Results, Primary and Repair Coatings (Phase I)

Hydrogen Embrittlement, ASTM F 519 Sustained Load Test in Air, Type 1a.1 AISI 4340 Notched Round Bars; SLT profile 75/200/5					
Coating	Replicate	FRACTURE STRENGTH (%)	TIME TO FAILURE (HRS)	Pass/Fail	Failure Analysis
LHE Cd	1	89.5%	202	Pass	
	2	92.1%	203		
	3	93.7%	203		
	4	91.9%	203		
IVD Al	1	92.9%	203	Pass	
	2	94.6%	203		
	3	94.2%	203		
	4	97.4%	204		
Alumiplate	1	91.6%	203	Pass	
	2	95.2%	203		
	3	96.0%	204		
	4	98.2%	204		
Sputtered Aluminum	1	81.40%	201	Pass	
	2	82.70%	201		
	3	84.70%	201		
	4	83.70%	201		
LHE Zn-Ni (IZ-C17)	1	92.0%	203	Pass	
	2	93.1%	203		
	3	92.8%	203		
	4	90.2%	202		
Zn-Ni (Boeing, acidic)	1	93.6%	203	Pass	
	2	90.7%	203		
	3	93.1%	203		
	4	94.8%	203		
Sn-Zn	1	75.3%	13.8	Fail	Bars #1 & 2: Single large IGF at surface
	2	75.2%	41.6		
	3	75.1%	108.3		
	4	75.1%	21.5		
	5	75.3%	7.1		
	6	75.0%	63		
	7	75.2%	7		
Brush LHE Cd	1	90.0%	202	Pass	
	2	91.7%	203		
	3	90.0%	202.8		
	4	95.0%	203		
Brush Sn-Zn	1	94.6%	203	Pass	
	2	92.7%	203		
	3	94.8%	203		
	4	95.3%	204		
Sermetel 249/273	1	96.4%	204	Pass	
	2	94.3%	204		
	3	94.9%	203		
	4	95.2%	203		
Brush Zn-Ni	1	89.2%	202	Fail	Bar #1: No IGF, fully ductile
	2	90.4%	202		IGF region initiating below the surface
	3	90.6%	203		
	4	75.2%	16.9		

Brush Zn-Ni came very close to passing the test but technically did not meet the criteria. One of four bars fractured well before 200 hours. The remaining three bars sustained 200 hours at 75% NFS, but none sustained an hour loaded at 90% NFS. Failure analysis was conducted on the bar that fractured prior to 200 hours and on one of the remaining three bars. As shown in Figure 49 the bar that fractured prior to 200 hours displayed intergranular fracture areas that did not originate at the surface. The other bar exhibited fully ductile fracture.

All four sputtered Al bars sustained 200 hours at 75% NFS and technically pass this test. However, upon step loading the bars fractured at roughly 81% to 85% NFS compared to the other coatings which generally sustained at least 90% NFS. By performing several additional tests, it was determined that the sputtering process had adversely affected the metallurgical temper of the test bars: Two extra sputtered Al bars were subjected to the constant rate tensile pull test, and the resultant notch tensile strength compared to that reported by the bar manufacturer for the lot. The sputter coated bars fractured 31.6 ksi and 48 ksi lower than the average for bare bars, indicating the steel strength was significantly compromised by the coating process. Two extra LHE Cd coated bars were also subjected to the constant rate tensile pull test to check the validity of the test. The Cd coated bars fractured 4.1 ksi and 5.7 ksi lower than the bare bars, which was well within the 10 ksi requirement. The Air Force C-17 program previously tested and verified that production landing gear components were not overheated and substrate steel was not degraded by the sputter coating process at Hill Air Force Base (per Boeing/C-17 Report # PTP-0113-Task F1, IWA #026406, dated 31 March 2005). The notched round bars used for this JTP test effort were coated by laboratory equipment uniquely designed for small round test coupons. The bars have significantly lower mass to surface area ratio than landing gear components and are thus more prone to overheating. Sputtered Al was not dropped from continued evaluation because the overheating issue was believed to be unique to small test bars and not large components. NAVAIR supplied additional round bars to the coater to validate the technique early in Phase II.

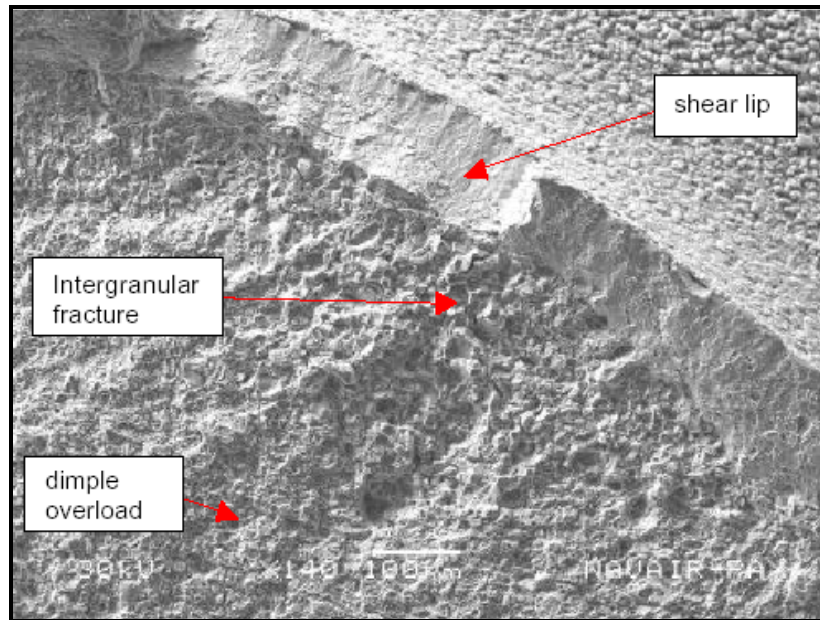


Figure 49. Fracture Surface of Brush Zn-Ni Bar That Failed at 16.9 Hours and 75% NFS

Hydrogen embrittlement testing was conducted again in Phase II as a quality assurance procedure to ensure reproducibility with Phase I results. The same test procedure was used. Four replicates of each coating were tested in Phase II, except for LHE Zn-Ni. All coatings passed the H-E requirements as shown in Table 64 below. The sputtered aluminum coating's Phase I results which had been determined to not be representative was processed at a lower power to limit temperature increase in the test bars and, as expected, the results reflected below are favorable: 97.1% average strength as compared to only 83.1% in Phase I.

Table 64. Hydrogen Embrittlement Reproducibility Test Results Coatings (Phase II)

Coating	Replicate	Fracture Strength (%)	Average Fracture Strength (%)	Time to Failure (hours)	Pass/Fail
LHE Cadmium	1	93.6%	92.7%	204	Pass
	2	93.7%		204	
	3	90.4%		204	
	4	93.1%		204	
Electroplated Al	1	92.6%	97.3%	203	Pass
	2	100.0%		204	
	3	97.3%		205	
	4	99.1%		205	
Sputtered Al	1	95.2%	97.1%	203	Pass
	2	95.1%		203	
	3	99.0%		205	
	4	99.1%		205	

6.1.3.6.2 Hydrogen Re-Embrittlement (HSS JTP 3.6.2)

Hydrogen re-embrittlement testing was conducted for all primary coatings that passed hydrogen embrittlement testing. In this test ASTM F 519 Type 1a.1, 4340 high strength steel notched round bars were loaded in tension while immersed in test fluids to determine whether the coating systems were resistant to in-service embrittlement. Four replicates of each coating were tested in each fluid. The fluids used were reagent water (ASTM D 1193 Type 2, 1-mega ohm), synthetic sea water (ASTM D 1141) and anti-freeze (three-to-one dilution propylene glycol to distilled water). About two to three milliliters of test fluid was contained in a small cup surrounding the bar notch. The fluid level extended approximately one quarter inch above and below the notch. Parafilm was wrapped from the top to bottom load frame grips to prevent evaporation of test fluid. Each coated bar was loaded individually for 24 hours at 45% of the notch fracture strength (NFS) established for uncoated bars of the same lot. The tensile load was subsequently increased by 5% per hour until bar fracture. ASTM F 519 stipulates a 150 hour sustained load duration rather than the incremental step load (ISL) used for Phase I of this effort. The JCAT chose to run the ISL method in Phase I as a screening test due to time and funding constraints, while still requiring the sustained load test prior to implementation decisions. It was reasoned that coatings that fail the 24 hour ISL test would fail the 150 hour sustained load test. Coatings that passed the Phase I ISL test were subsequently evaluated in the longer sustained load test in Phase II. The acceptance criteria for the ISL test were that the average load and time to fracture be greater than or equal to LHE Cd when tested in 1 mega ohm reagent water. Photographs of the test equipment and close-ups of the fluid containing cup around the specimen are shown in Figure 50. Phase I test results as originally published are shown in Table 65.



Figure 50. Hydrogen Re-Embrittlement Test Equipment

Table 65. Hydrogen Re-Embrittlement Test Results (Phase I)

Hydrogen Re-Embrittlement, ASTM F 519 Annex 3 Rising Step Load Test in Operational Fluids, Type 1a.1 AISI 4340 Notched Round Bars; RSL profile 45/24/5						
Coating	Fluid	Replicate	Fracture Strength (%)	Load at Failure (LBS)	Time to Failure (HRS)	Pass/Fail*
LHE Cd	3:1 Propylene Glycol/Distilled Water	1	90.1%	8478.2	32	Control
		2	89.9%	8467.5	32	
		3	90.2%	8489.9	32.2	
		4	90.0%	8475.6	32.1	
		AVG	90.1%	8477.8	32.1	
		STDEV			0.1	
	ASTM D 1141 Synthetic Sea Water	1	90.6%	8533.2	32	
		2	90.0%	8475.6	32	
		3	55.0%	5178.6	25.1	
		4	75.2%	7075.3	29.8	
		AVG	77.7%	7315.7	29.7	
		STDEV			3.3	
	ASTM D 1193 Type 2, 1-mega ohm reagent water	1	85.3%	8026.5	31.1	
		2	65.3%	6145.5	27	
		3	80.0%	7532.3	30	
		4	65.1%	6127	27.1	
		AVG	73.9%	6957.8	28.8	
		STDEV			2.1	
Alumiplate	3:1 Propylene Glycol/Distilled Water	1	95.1%	8956.7	33	Pass
		2	95.0%	8942.4	33	
		3	95.0%	8945.8	33	
		4	95.2%	8962.5	33	
		AVG	95.1%	8951.9	33.0	
		STDEV			0.0	
	ASTM D 1141 Synthetic Sea Water	1	95.2%	8965.2	62	
		2	90.3%	8497.7	32.8	
		3	95.1%	8952.1	33.2	
		4	95.1%	8949.5	33	
		AVG	93.9%	8841.1	33.0	
		STDEV			0.2	
	ASTM D 1193 Type 2, 1-mega ohm reagent water	1	95.3%	8968.4	33.2	
		2	94.3%	8878.9	33	
		3	95.2%	8960.2	33	
		4	95.1%	8952.4	34	
		AVG	95.0%	8940.0	33.3	
		STDEV			0.5	
Zn-Ni (Dipsol LHE)	3:1 Propylene Glycol/Distilled Water	1	95.00%	8668.2	33	Marginal Fail
		2	93.10%	8488.6	33	
		3	93.90%	8562.6	33	
		4	90.00%	8475	32	
		AVG	93.0%	8548.6	32.8	
		STDEV			0.5	
	ASTM D 1141 Synthetic Sea Water	1	45.00%	4107.3	0.7	
		2	65.10%	5941.6	27.4	
		3	60.00%	5473.1	26.1	
		4	60.20%	5491.1	26.5	
		AVG	57.6%	5253.3	20.2	
		STDEV			13.0	
	ASTM D 1193 Type 2, 1-mega ohm reagent water	1	90.30%	8235.4	32	
		2	60.30%	5496.3	26.2	
		3	80.00%	7298.2	30.1	
		4	50.20%	4579.5	24.1	
		AVG	70.2%	6402.4	28.1	
		STDEV			3.6	
IVD Aluminum	3:1 Propylene Glycol/Distilled Water	1	80.5%	7577	30.8	Fail
		2	75.2%	7081.5	29.4	
		3	80.5%	7581.5	30.2	
		4	75.1%	7069.8	29.2	
		AVG	77.8%	7327.5	29.9	
		STDEV			0.7	
	ASTM D 1141 Synthetic Sea Water	1	50.3%	4735.1	24.1	
		2	50.3%	4731.8	24.1	
		3	55.0%	5178.4	25.1	
		4	55.0%	5178.2	25.1	
		AVG	52.7%	4955.9	24.6	
		STDEV			0.6	
	ASTM D 1193 Type 2, 1-mega ohm reagent water	1	65.1%	6127.1	27.8	
		2	45.2%	4254.4	13.9	
		3	45.0%	4237.1	18	
		4	45.3%	4260.9	13.2	
		AVG	50.2%	4719.9	18.2	
		STDEV			6.7	

Table 65 (con't). Hydrogen Re-Embrittlement Test Results (Phase I)

Coating	Fluid	Replicate	FRACTURE STRENGTH (%)	LOAD AT FAILURE (LBS)	TIME TO FAILURE (HRS)	Pass/Fail*	Failure Analysis and Testing Notes
Sputtered Aluminum	3:1 Propylene Glycol/Distilled Water	1	65.0%	6121.7	27.1	Fail	Irregular IGF with mixed ductile/faceted overload
		2	84.4%	7945	31		
		3	80.0%	7531.7	30		
		4	80.1%	7543.6	30		
		AVG	77.4%	7285.5	29.5		
		STDEV			1.7		
	ASTM D 1141 Synthetic Sea Water	1	45.2%	4259.2	0.1		Single large IGF initiating at surface
		2	45.1%	4250.4	0.1		Irregular IGF with mixed ductile/faceted overload
		3	50.0%	4708.9	24.1		Multiple large IGF initiating at surface
		4	55.5%	5222.5	25.7		Irregular IGF with mixed ductile/faceted overload
		AVG	49.0%	4610.3	12.5		
		STDEV			14.3		
	ASTM D 1193 Type 2, 1-mega ohm reagent water	1	45.2%	4257.3	6.3		Single large IGF initiating at surface
		2	50.2%	4728.8	24.5		Single large IGF initiating at surface
		3	50.0%	4707.3	24.2		Single large IGF initiating at surface
		4	45.3%	4261.7	1.5		Single large IGF initiating at surface
		AVG	47.7%	4488.8	14.1		
		STDEV			12.0		
Zn-Ni (Boeing, acidic)	3:1 Propylene Glycol/Distilled Water	1	55.0%	5178.3	25.6	Fail	Single large IGF initiating at surface
		2	45.0%	4238.7	0.3		Single large IGF initiating at surface
		3	60.0%	5652.7	26.1		Single large IGF initiating at surface
		4	70.5%	6633.9	28.1		Single large IGF initiating at surface
		AVG	57.6%	5425.9	20.0		
		STDEV			13.2		
	ASTM D 1141 Synthetic Sea Water	1	45.1%	4248.6	0.1		Single large IGF initiating at surface
		2	45.0%	4238.7	0.1		Multiple large IGF initiating at surface
		3	50.0%	4707.4	24		Multiple large IGF initiating at surface
		4	45.3%	4262.2	4.3		Single large IGF initiating at surface
		AVG	46.4%	4364.2	7.1		
		STDEV			11.4		
	ASTM D 1193 Type 2, 1-mega ohm reagent water	1	50.5%	4752.4	25		Multiple large IGF initiating at surface
		2	45.2%	4250.7	1.2		Single large IGF initiating at surface
		3	45.0%	4238.1	0		Single large IGF initiating at surface
		4	45.0%	4237.1	0.1		Single large IGF initiating at surface
		AVG	46.4%	4369.6	6.6		
		STDEV			12.3		
* Acceptance Criteria: average load and time to fracture greater than or equal to LHE Cd when tested in 1-mega ohm reagent water.							
IGF = Intergranular Fracture							

In the Phase I H-RE testing AlumiPlate™ performed best compared to all coatings including LHE Cd. The AlumiPlate™ coating is relatively dense compared to IVD-Al and sputter coatings, and its excellent performance in this test may be partly due to its barrier properties. Figure 51 visually depicts the relative difference of coating densities among AlumiPlate™, IVD aluminum and sputtered aluminum. LHE Zn-Ni also performed well with only a slight decrease in performance compared to LHE Cd. IVD-Al performed slightly better than Sputtered Al, and both performed significantly worse than LHE Cd. Sputtered Al is a denser coating than IVD Aluminum and was expected to perform better than IVD Al, although the Phase I sputtered Al results were rated as a 'Fail' due to the overheating issue discussed. During the test results discussion a JCAT member noted failures of C-5 base nuts coated with IVD Al that led to a policy at Hill Air Force Base that now requires IVD Al to be subsequently painted. Boeing's acidic Zn-Ni coating provided the least protection and was voted to be dropped from further consideration in this test effort. The acidic Zn-Ni coating is, however, being implemented on a case by case basis on high strength steel components on Boeing commercial aircraft.

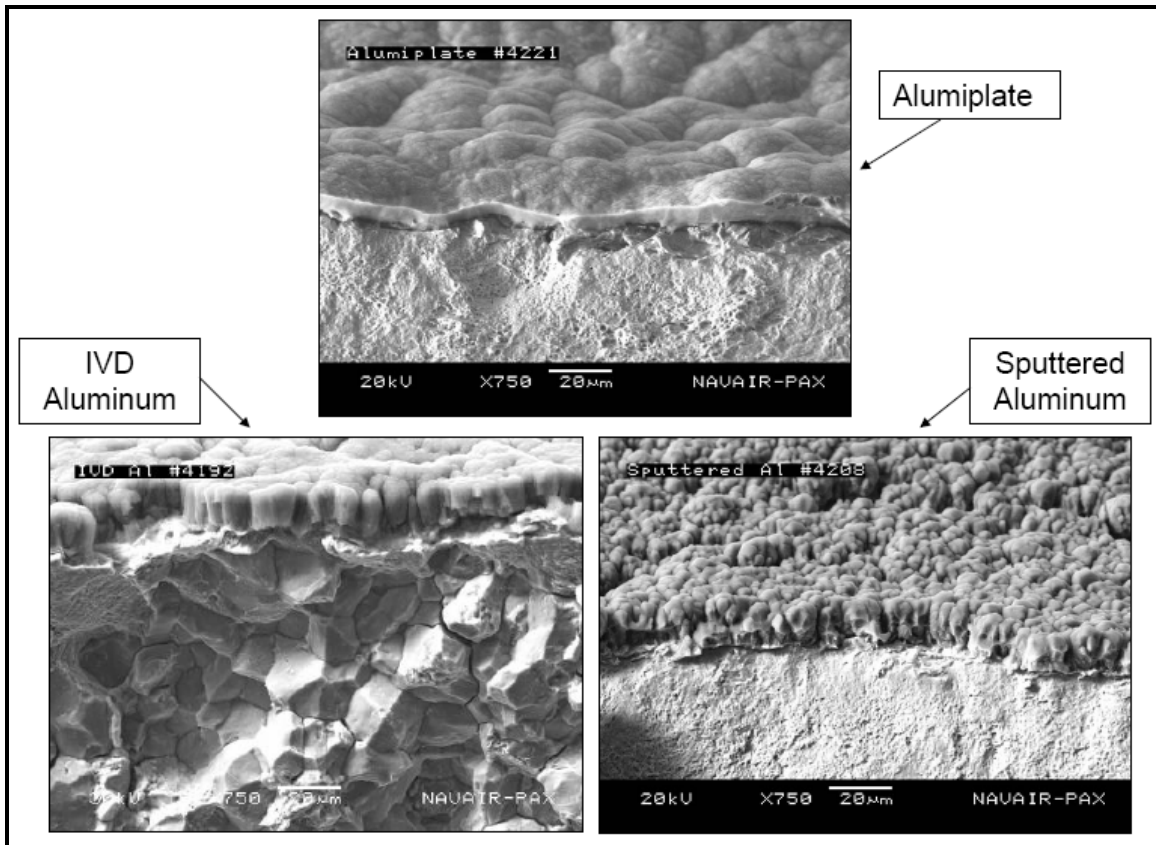


Figure 51. Visual Depiction of Aluminum Coating Density for Different Deposition Technologies

IVD Al performed worse than LHE Cd for all three test fluids. The reagent water test results for LHE Cd and IVD Al were consistent with Boeing historical test data. In the reagent water test LHE Cd failed at 6,958 pounds load at 28.8 hours and IVD Al failed at 4,720 pounds load at 18.2 hours (average of four specimens). During the test results discussion it was suggested that alternative coatings should meet or exceed the performance of IVD Al rather than LHE Cd. This is something to consider only if quantitative field data covering representative service environments proves equivalent or better field performance of IVD Al compared to LHE Cd. Such field data has not been formally reported to date, and if or when such data is presented then contradictory cases like the C-5 base nuts discussed above must also be considered.

Failure analysis of tested specimens was conducted to identify whether the bars had experienced ductile fracture or failed due to hydrogen embrittlement. For each coating and test fluid set at least one bar was examined. In cases where all four bars had similar results only one bar was examined and was considered representative of the set. In cases where results varied among the set all four bars were examined. For LHE Cd the test results were similar for all three fluids and only one bar was examined from each set. The synthetic sea water bar exhibited fully ductile fracture with some facets potentially indicating brittle fracture, and the reagent water and propylene glycol/water bars both exhibited single large intergranular fracture areas initiated at the surface. The AlumiPlate™ bars also had similar test results, and bars from all three fluids exhibited intergranular fracture. The test results for LHE Zn-Ni were slightly scattered for the

sea water and reagent water bars. All LHE Zn-Ni bars that were examined exhibited intergranular fracture. The IVD Al test results were similar for propylene glycol and sea water bars but scattered for the reagent water bars. All IVD Al bars that were examined exhibited intergranular fracture. The sputtered Al test results were similar for propylene glycol but scattered for the sea water and reagent water bars. All sputtered Al bars that were examined exhibited intergranular fracture. Test results for the acidic Zn-Ni coating were scattered for all three fluids. All acidic Zn-Ni bars were examined and all bars exhibited intergranular fracture. The full failure analysis report is located in Appendix D, and includes selected photographs of the fracture surfaces.

Phase II H-RE Results: Selected H-RE tests were performed in Phase II to establish reproducibility trends for each coating dependent on availability of additional test specimens. Results are presented in Table 66. Consistent with Phase I, re-embrittlement test results for the AlumiPlate™ coating were best in Phase II with fracture load values well into the 90% range for each test fluid environment. In Phase II, LHE Zn-Ni performed much better in reagent water (93% vs. 70.2% in Phase I) yet about the same in synthetic seawater (~58% average). The favorable reagent water result fracture loads in Phase II would have qualified as a ‘Pass’ rating in the JTP acceptance criteria except that two additional specimens needed to be run to meet the 4 bar requirement (these two bars had been inadvertently run in synthetic seawater). In the seawater test, two specimens failed prior to 150 hours in Phase II, whereas only one did in Phase I. Tests were not performed in the propylene glycol for LHE Zn-Ni. One interesting note is that the best performing specimen in the seawater test had been mistakenly loaded in air to 75% NFS (180 hours), then re-started at 45% in seawater to complete that dataset. This specimen failed at 80.3% NFS which was the highest of the 8 round bars tested in Phases I/II.

Sputtered Al was fully re-tested for re-embrittlement characteristics in Phase II due to the process change which was required to avoid overheating of the ¼ inch diameter round bar specimens (ASTM F 519, Type 1a.1). This process change will not be required for full size components so the data was generated for comparative purposes. While results had improved for H-E testing as expected (Sec. 6.1.3.6.1), results for H-RE were fairly similar to those obtained in Phase I. Average fracture strength for sputtered Al was statistically about the same in reagent water (48.9% vs. 47.7%) meaning it did not earn a ‘Pass’ rating in Phase II. The coating performed slightly better in propylene glycol in Phase II (85.3% vs. 77.4%) but slightly lower in synthetic seawater (45.2% vs. 49%), so overall it performed very similar to Phase I. In reagent water, two sputtered Al bars failed before the end of the 45% hold in each test phase. In synthetic seawater, the Phase II specimens all fractured within 1 hour at the 45% static load, while two of four specimens in Phase I had also failed within 1 hour, and the other two failed at slightly higher values (50.0% and 55.5% NFS). This test variability seems to represent normal statistical variation.

The incremental step load hydrogen re-embrittlement test is an efficient method to screen new coatings for in-service embrittlement performance, however, it is recommended that the ASTM F 519 Annex 5 150-hr sustained load test in service fluids be conducted as further quality control prior to implementation decisions. In addition, users may consider conducting re-embrittlement tests using intentionally scribed/damaged coatings. Components in the field are often scratched and thus testing specimens in a scratched condition makes sense. One way to simulate a scratch

without actually scratching the specimen is to mask the notch area of a Type 1a.1 round bar, for example. NAVAIR Pax River has done this for other projects recently by tightly tying a thin, Teflon®-based thread in the notch prior to coating application.

Table 66. Hydrogen Re-Embrittlement Results Comparison (Phase II vs. Phase I) for Coatings Tested in Both Phases

Test Phase →			Phase II Results			Phase I Results (1)		
Coating	Test Fluid	Rep.	Fracture Strength (%)	Average Fracture Strength (%)	Time to Failure (hours)	Fracture Strength (%)	Average Fracture Strength (%)	Time to Failure (hours)
LHE Zn-Ni	ASTM D1141 Sea Water	1	60.3%	57.7%	153	45.0%	57.6%	0.7
		2	45.0%		13	65.1%		27.4
		3	80.3% (2)		157	60.0%		26.1
		4	45.0%		< 1	60.2%		26.5
	ASTM D1193 Reagent Water	1	90.5%	93.0%	157	90.3%	70.2%	32.0
		2	95.4%		160	60.3%		26.1
		3	--		--	80.0%		30.1
		4	--		--	50.2%		24.1
Electroplated Al	ASTM D1141 Sea Water	1	99.8%	99.0%	160	Note (4)	93.9%	
		2	98.2%		160 (3)			
	3:1 propylene glycol	1	98.8%	95.8%	160		95.1%	
		2	92.7%		159			
	ASTM D1193 Reagent Water	1	93.2%	95.7%	159		95.0%	
		2	98.2%		157			
Sputtered Al	ASTM D1141 Sea Water	1	45.0%	45.2%	0.1	45.2%	49.0%	0.1
		2	45.2%		0.1	45.1%		0.1
		3	45.0%		0.1	50.0%		24.1
		4	45.4%		0.9	55.5%		25.7
	3:1 propylene glycol	1	90.2%	85.3%	158.9	65.0%	77.4%	27.1
		2	80.5%		136.0	84.4%		31.0
		3	90.0%		158.8	80.0%		30.0
		4	80.4%		156.6	80.1%		30.0
	ASTM D1193 Reagent Water	1	45.1%	48.9%	42.4	45.2%	47.7%	6.3
		2	50.2%		151	50.2%		24.5
		3	45.0%		81.8	50.0%		24.2
		4	55.4%		151.4	45.3%		1.5

Notes: (1) The test profile in Phase I was shorter in duration than Phase II (24 hour vs. 150 hour initial hold at 45%); (2) Sample loaded to 75% NFS in air for 180h, then re-started at 45% in sea water environment; (3) Machine experienced a power failure; the sample remained under load and the test was restarted at ~96 h; (4) Phase I averages provided for AlumiPlate represent the average of four specimens for each fluid (all individual specimens were >90%).

6.1.3.7.1 Repairability (HSS JTP Sec. 3.7.1)

This section contains results of the testing on cadmium alternative repair coatings, two of which were applied by selective (brush) plating and one that is more like a paint. Repair coatings were subjected to the sub-set of JTP testing which was described in Sec. 5.1.3.7.1.

Appearance

Two of the three candidate repair coatings, as well as the baseline coating, achieved a pass rating. The brush plated Sn-Zn coating achieved a “fail” rating, due in part to the observation of a dark brown area through the center of the panel. The observations from the appearance evaluation of the repair coatings are located in Table 67.

Table 67: Appearance of Repair Coatings

Coating	Appearance Results
Brush Plated Cd (Baseline) - Boeing	Coating is continuous but not uniform, showing swirls from processing; coating is smooth, adherent, and free from blisters, pits, excessive powder, and contamination
Brush Plated Zn-Ni - Boeing	Coating is not continuous or uniform; coating is adherent, but rough, with excessive powder and possible rust spots
Brush Plated Sn-Zn - Boeing	Coating is continuous but not uniform, with a dark brown area through the center of the panel; the coating is smooth and adherent, but has excessive powder
Sprayed Al-Ceramic (SermeTel[®] 249/273) – Boeing	Coating is continuous and uniform, smooth, adherent, and free from pits, blisters, excessive powder, and contamination

Bend Adhesion

The results of bend adhesion testing of the repair coatings are listed in Table 68. In addition, Figure 52 contains representative photos of the test specimens after testing. Bend adhesion test results for the repair coatings were similar to those obtained in Phase I (again only tested on low alloy 4130 steel). Brush plated Cd exhibited significant adhesion loss after 1.5 bend cycles (second tensile cycle for the coating) as shown in Fig. 52(a). Application of a sharp blade easily removed most of the coating. The brush Zn-Ni panels performed well on all specimens, with only slight coating removal at the edges of the specimens. The bulk of the area did not have visually apparent cracks developing until 8-9 bend cycles, and the deposit was adherent when challenged with a blade after substrate failure (Fig. 52(b)). Brush Sn-Zn performed well on all specimens; one representative panel is shown in Figure 52(c). The SermeTel[®] coating exhibited adhesion failure after 2-3 cycles, furthermore light brushing with a fingernail removed the coating across the full width of one specimen out of three (right side of Fig. 52(d)), while the other two exhibited removal closer to the edges (left side of Fig. 52(d)).

Table 68: Bend Adhesion Results for Repair Coatings

Coating	Replicate	Cycles to Fracture	Comments	Pass/Fail
Brush Plated Cd	1	2-3	Significant coating adhesion loss after 1.5-3 bend cycles	Fail
	2	2		
	3	2-3		
Brush Plated Zn-Ni	1	13	Coating cracks apparent by 8-9 cycles, but coating cannot be lifted with sharp blade	Pass
	2	11		
	3	12		
Brush Plated Sn-Zn	1	10		Pass
	2	17		
	3	14		
Al-Ceramic (SermeTel [®] 249/273)	1	2	Coating failure near edges, although failure was across full width of 2 specimens when brushed lightly	Fail
	2	2-3		
	3	3		



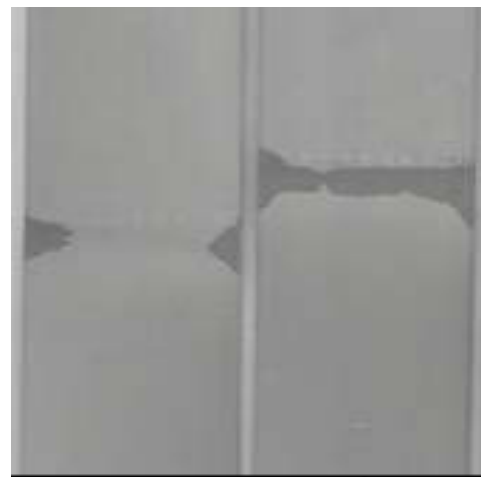
(a) Brush Plated Cd



(b) Brush Plated Zn-Ni



(c) Brush Plated Sn-Zn



(d) Al-Ceramic (SermeTel[®] 249/273)

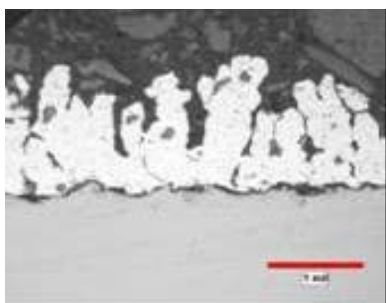
Figure 52. Representative Photos of Bend Adhesion Results for Repair Coatings

Thickness

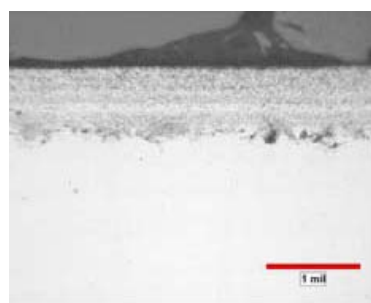
Table 69 contains the results of the thickness measurements with representative cross sectional micrographs shown in Figure 53.

Table 69. Coating Thickness Results for Repair Coatings

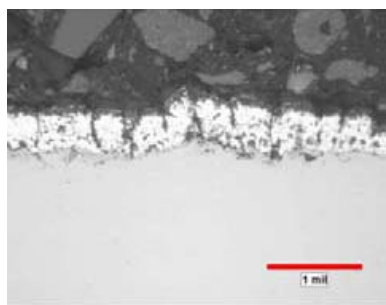
Coating	Thickness Measurements (mil)					
	Reading → #1	#2	#3	#4	#5	Avg.
Brush Plated Cd	1.54	1.57	1.28	1.14	1.19	1.34
Brush Plated Zn-Ni	1.16	0.93	0.78	0.79	0.77	0.89
Brush Plated Sn-Zn	0.48	0.53	0.57	0.51	0.41	0.50
SermeTel [®]	1.47	1.37	1.51	1.45	1.28	1.42



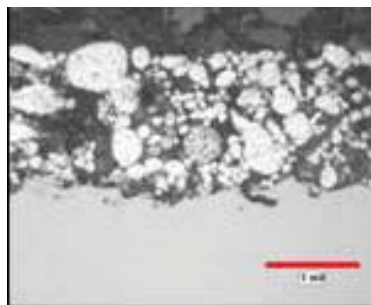
(a) Brush Plated Cd



(b) Brush Plated Zn-Ni



(c) Brush Plated Sn-Zn



(d) SermeTel[®]

**Figure 53: Cross-sectional Images of Coating for Thickness Measurement, 500X
(micron bar shows 1 mil = 25.4 μ m)**

If plating to the specification, all coatings including brush cadmium exceeded the specification. The Sn-Zn coating was closest to the 0.3–0.5 mil thickness requirement. Brush cadmium thickness should probably be considered an acceptable baseline since actual field-applied thickness variability is not known.

Unscribed Corrosion Resistance

Table 70 lists the results of the unscribed corrosion resistance test. Photos of the panels at the conclusion of testing are located in Figure 54.

Table 70. Unscribed Corrosion Resistance Results for Repair Coatings

Coating	Panel #	First Appearance of Corrosion/Observation	Time of Termination/Observation	Rating at Test Termination
Brush Plated Cd	1	No rust	3000 hours/No rust	10
	2	No rust	3000 hours/No rust	10
	3	No rust	3000 hours/No rust	10
Brush Plated Zn-Ni	1	No rust	3000 hours/Sacrificial coating breakdown/no rust	9
	2	No rust	3000 hours/Sacrificial coating breakdown/no rust	9
	3	No rust	3000 hours/Sacrificial coating breakdown/no rust	9
Brush Plated Sn-Zn	1	72 hours/Chromate depletion	3000 hours/Chromate depletion and pin holes	8
	2	72 hours/Chromate depletion	3000 hours/Chromate depletion and pin holes	8
	3	72 hours/Chromate depletion	3000 hours/Chromate depletion and pin holes	8
Al-Ceramic (SermeTel®)	1	24 hours/Pinhole rust spots through coating	500 hours/Excessive rust	0
	2	24 hours/Pinhole rust spots through coating	500 hours/Excessive rust	0
	3	24 hours/Pinhole rust spots through coating	500 hours/Excessive rust	0



(a) Brush Plated Cd (3000 hours)



(b) Brush Plated Zn-Ni (3000 hours)



(c) Brush Plated Sn-Zn (3000 hours)



(d) Al-Ceramic (SermeTel®) (500 hours)

Figure 54. Photos of Unscribed Corrosion Resistance Panels for Repair Coatings

The brush plated cadmium coatings showed excellent corrosion resistance at 3000 hours with little or no red rust present in the center test areas of the panel, and was thus ranked highest in this test. The LHE Zn-Ni coating also exhibited good performance with no steel corrosion although there was breakdown of the sacrificial coating during the test. The brush Sn-Zn coating showed early signs of chromate depletion within one week, and by the end of the test there were some corrosion pinholes in the coating. The spray-applied SermeTel® 249 coating performed similar to the primary coatings sputtered Al and also some of the IVD-Al panels with early rust formation causing test termination at about 500 hours.

Scribed Corrosion Resistance

Table 71 lists the results of the scribed corrosion resistance evaluation of the repair coatings. In addition, Figure 55 contains photos of the panels after testing. The results for the scribed panels are very similar to the results for the unscribed panels, with the exception of the Sn-Zn panels. These panels developed rust in the scribe and were pulled from testing due to excessive rust at 500 hours.

Table 71. Scribed Corrosion Resistance Test Results for Repair Coatings

Coating	Panel #	First Appearance of Corrosion/Observation	Time of Termination/Observation	Rating at Test Termination
Brush Plated Cd	1	No rust	3000 hours/No rust	9
	2	No rust	3000 hours/No rust	9
	3	No rust	3000 hours/No rust	9
Brush Plated Zn-Ni	1	No rust	3000 hours/Sacrificial coating breakdown/no rust	9
	2	No rust	3000 hours/Sacrificial coating breakdown/no rust	7
	3	No rust	3000 hours/Sacrificial coating breakdown/no rust	7
Brush Plated Sn-Zn	1	168 hours/Rust in scribe	500 hours/Excessive Rust	9 at scribe
	2	168 hours/Rust in scribe	500 hours/Excessive Rust	9 at scribe
	3	168 hours/Rust in scribe	500 hours/Excessive Rust	9 at scribe
Al-Ceramic (SermeTel[®])	1	24 hours/Pinhole rust spots through coating	500 hours/Excessive rust	0
	2	24 hours/Pinhole rust spots through coating	500 hours/Excessive rust	0
	3	24 hours/Pinhole rust spots through coating	500 hours/Excessive rust	0



(a) Brush Plated Cd (3000 hours)



(b) Brush Plated Zn-Ni (3000 hours)



(c) Brush Plated Sn-Zn (500 hours)



(d) Al-Ceramic (SermeTel[®]) (500 hours)

Figure 55. Photographs of Scribed Corrosion Resistance Panels for Repair Coatings

Hydrogen Embrittlement – Reproducibility

Table 72 lists the results of the H-E test, which include the fracture strength and time to failure.

Table 72: Hydrogen Embrittlement/Reproducibility Test Results for the Repair Coatings

Coating	Replicate	Fracture Strength (%)	Average Fracture Strength	Time To Failure (Hours)	Pass/Fail
Brush Plated Cadmium	1	95.1%	96.3%	203	Pass
	2	95.0%		203	
	3	97.5%		204	
	4	97.6%		204	
Brush Plated Zn-Ni	1	92.5%	93.8%	203	Pass
	2	93.0%		203	
	3	94.6%		203	
	4	94.9%		203	
Brush Plated Sn-Zn	1	100.0%	98.1%	205	Pass
	2	96.1%		205	
	3	97.1%		205	
	4	99.3%		205	
Al-Ceramic (SermeTel [®])	1	97.6%	96.0%	204	Pass
	2	94.9%		203	
	3	95.1%		204	
	4	96.4%		204	

For the repair coatings, all four brush Zn-Ni bars tested passed with average fracture strength of 93.8%. The brush Sn-Zn coating had the highest average strength at 98.1%, while the brush Cd and SermeTel[®] 249 coatings were 96.3% and 96.0%, respectively.

Mixed results were obtained for the repair coatings. Brush plated Zn-Ni performed best overall, although it received a failing rating for coating appearance due to excessive surface roughness. Brush plated Sn-Zn was ranked second, with failing results noted in corrosion tests. The SermeTel[®] sprayed Al ceramic coating performed worst of the repair coatings, failing both corrosion and adhesion tests. The results indicate that while brush plated Zn-Ni performed best, additional testing and evaluation is required to identify and confirm a suitable replacement for brush plated cadmium.

6.1.4.0 Navy Added Corrosion Testing Results

Since some of the Navy added test specimens were obtained from different sources than the umbrella ESTCP WP-0022 project, these differences will be described here. The Navy add on tests did not include the magnetron sputtered aluminum coating, instead one additional electroplated zinc-nickel coating, nominally Zn-6Ni, was included as a control to the LHE Zn-Ni IZ-C17 process being investigated under the WP-0022 project.

For the Navy added testing the high-strength test alloy was chosen to be AISI 4340 steel for the SCC and fatigue tests, while 4130 alloy steel was used for the cyclic SO₂ corrosion tests. SCC and fatigue specimens were machined from a tempered 4340 steel plate of 3.8 x 15 x 30 cm. Round rod specimens were prepared in a lathe (fatigue) and square bar specimens (SCC) by means of electric discharge machining (EDM). Surfaces were manually polished with 120 grit emery cloth after machining to remove the EDM layer. Specimen dimensions were: round tension test specimens of diameter 6.4 mm and gage length 25.4 mm in longitudinal (L-) orientation; round hourglass fatigue test specimens of minimum diameter 6.4 mm and gage length 49.3 mm in L-orientation; and square bar stress corrosion cracking test specimens of 10 x 10 x 50.8 mm with a central v-notch of angle 60°, tip radius 0.15 mm and depth 5 mm in L-T orientation.

Test coatings applied to the test specimens were as follows: The machined and polished specimens were electroplated with AlumiPlate™, cadmium, Zn-6Ni and Zn-13Ni, and also vacuum coated with IVD-Al. A summary of each coating process is provided below.

- AlumiPlate™: High purity Al was electroplated on the specimens per MIL-DTL-83488 Class 2, Type 2. Specimens were subjected to chromate treatment per MIL-DTL-5541, Type 1, Class 1A. Specimens were coated by AlumiPlate Inc.
- IVD Al: Al was ion vapor deposited on the specimens per MIL-DTL-83488 Class 2, Type 2. Specimens were subjected to chromate treatment per MIL-DTL-5541, Type 1, Class 1A. Specimens were coated by the Navy's Fleet Readiness Center Southwest (FRC-SW, North Island).
- Cadmium: Cd was electroplated on the specimens per MIL-STD-870. Specimens were subjected to the standard hydrogen embrittlement relief, and subsequent chromate treatment per MIL-DTL-5541, Type 1, Class 1A. Specimens were coated by FRC-SW.
- Zn-6Ni: Zn-6Ni alloy was electroplated on the specimens per AMS 2417G, Type 2, alkaline Zn-Ni Plating (IZ-260 product) for 30 minutes at room temperature. Then the specimens were baked at 375°F for 23 hours and chromate conversion coated per MIL-DTL-5541, Type 1, Class 1A. Specimens were coated by Dipsol of America.
- Zn-13Ni: Zn-13Ni alloy was electroplated on the specimen per IZ-C17 Alkaline Zn-Ni Plating Process (3 A/dm² at room temperature for 45 min). Specimens were subjected to the standard hydrogen embrittlement relief, and subsequent chromate treatment per manufacturer recommendation. Specimens were coated by Dipsol of America.

Coated specimens were examined to verify that coatings covered the specimens fully, including the notch. The coating thickness was measured from the backscattered electron image of specimen cross-sections. Chemical composition was determined using electron microprobe with wavelength dispersive X-ray spectrometer, under conditions of 15 keV, 30 nA, 1 µm spot size and 20 second counting time. In addition, the residual stresses in the coating and substrate were measured with the aid of X-ray diffraction. For reference, the residual stress profile in a bare specimen was also determined.

6.1.4.1.1 Unscribed Cyclic SO₂ Salt Spray (Fog) Corrosion Resistance (HSS JTP Sec. 4.1.1)

In the acidified salt fog environment (ASTM G 85, Annex 4) the three types of aluminum coatings performed better than cadmium and LHE Zn-Ni. In the unscribed condition, cadmium showed significant failure by four days (96 h) in the sulfur dioxide environment (Fig. 56a). The LHE Zn-Ni coating performed substantially better than cadmium in this study; however, some red rust was apparent after one week (Fig. 56b). After two weeks of exposure, the LHE Zn-Ni panels were mostly corroded and were judged to have performed roughly twice as good as cadmium in this environment. IVD-Al exhibited red rust on one of three panels prior to the 3 week (504 hour) inspection, and is shown at the 4 week (668 h) inspection in Figure 56c. The AlumiPlate[™] coating had the best appearance of the alternatives when evaluated after 668 hours (Fig. 56d), where two of three panels are significantly better than either of the other aluminum coatings tested. The sputtered aluminum coating was fairly similar to IVD-Al in performance after factoring out the bottom portions of the panels, which failed prematurely due to inferior protection of the back surface (Note: IVD-Al and AlumiPlate[™] coated panels had aluminum coatings applied on both sides of the panels, while the line-of-sight sputtered coating had been applied to only the front surface. Therefore, premature sputtered Al coating failure initiated from the bottom edges of the panels which were not covered by the aluminum coating). A graphic representation of panel ratings with test time is provided in Fig. 57.



(a) Cadmium (96 hr)



(b) LHE Zn-Ni (168 hr)



(c) IVD Aluminum (668 hr)



(d) AlumiPlate™ (668 hr)



(e) Sputtered Al (668 hr)

Figure 56. Unscribed Cyclic SO₂ Salt Fog Corrosion Resistance

The graph in Figure 57 shows how the ratings by visual examination of the panels in the chamber changed over time. From the graph, it is clear that all coatings performed better than Cd, with each of the Al coatings also performing better than the LHE Zn-Ni coating.

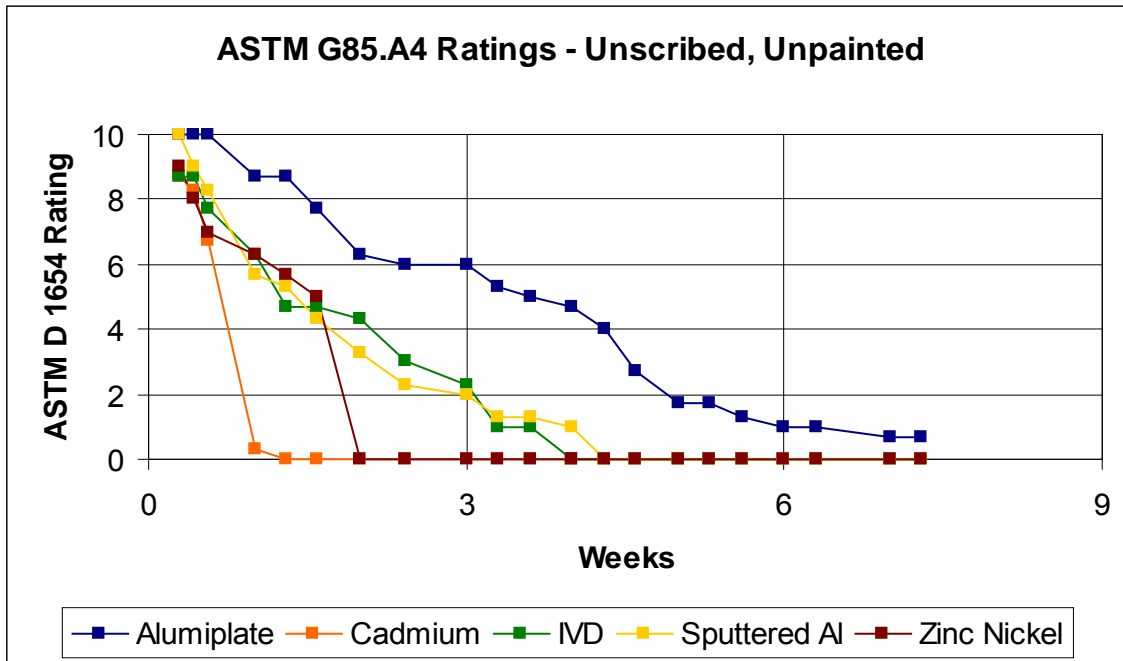


Figure 57. Unscribed Cyclic SO₂ Salt Fog Corrosion Resistance (Ratings vs. Time)

6.1.4.1.2 Scribed Cyclic SO₂ Salt Spray (Fog) Corrosion Resistance (HSS JTP Sec. 4.1.2)

Two out of three scribed cadmium plated panels exhibited red rust by 96 hours (Fig. 58a). At 168 hours, the LHE Zn-Ni panels also exhibited red rust, most noticeable in top portions of the scribes (Fig. 58b). After two weeks the LHE Zn-Ni panels had failed and were similar in appearance to how the cadmium panels had appeared after one week. After two weeks (336 h) the IVD-Al panels contained some red rust in the scribes as well as in the field area of one panel (Fig. 58c). The AlumiPlate™ and sputtered Al coatings both resisted red rust in the scribes at 668 h (Fig. 58d and 58e, respectively). Corrosion performance ratings over a time span of 7 weeks are shown in Figure 59, where the higher performance of the aluminum coatings as a group is evident.



(a) Cadmium (96 hr)



(b) LHE Zn-Ni (168 hr)



(c) IVD Aluminum (336 hr)



(d) AlumiPlate™ (668 hr)



(e) Sputtered Al (668 hr)

Figure 58. Scribed Cyclic SO₂ Salt Fog Corrosion Resistance

Figure 59 contains a graph of the results of SO₂ corrosion resistance for the scribed panels, which are very similar to the results seen for the unscribed panels, with all alternative coatings performing better than the Cd-plated panels.

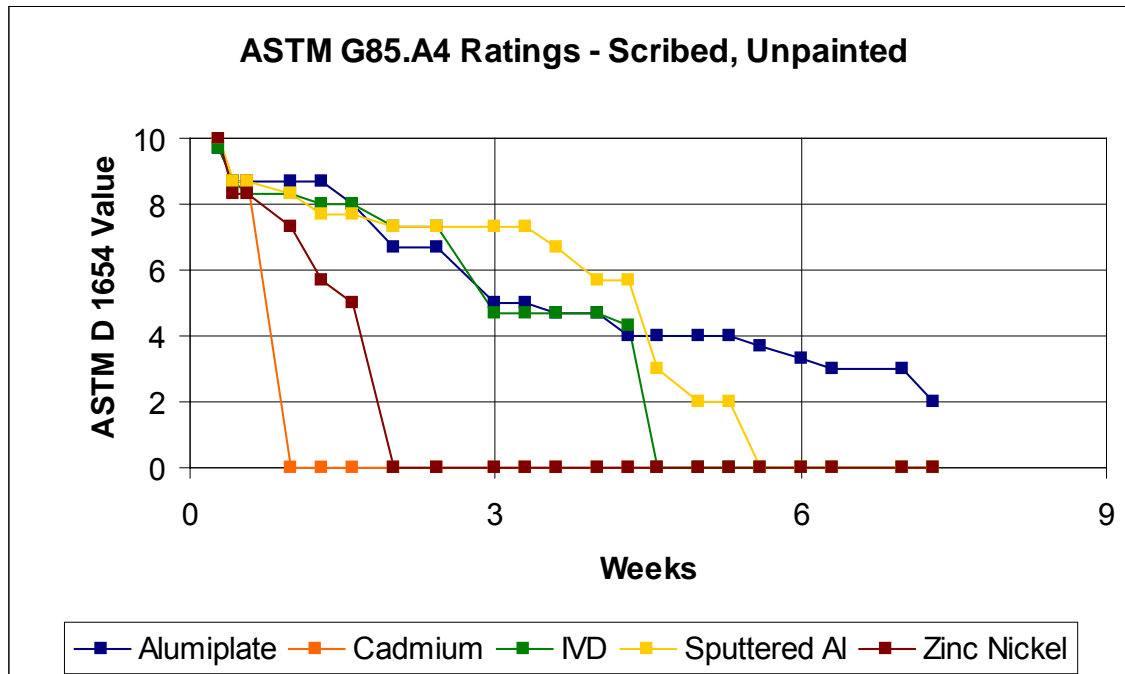


Figure 59. Scribed Cyclic SO₂ Salt Fog Corrosion Resistance (Ratings vs. Time)

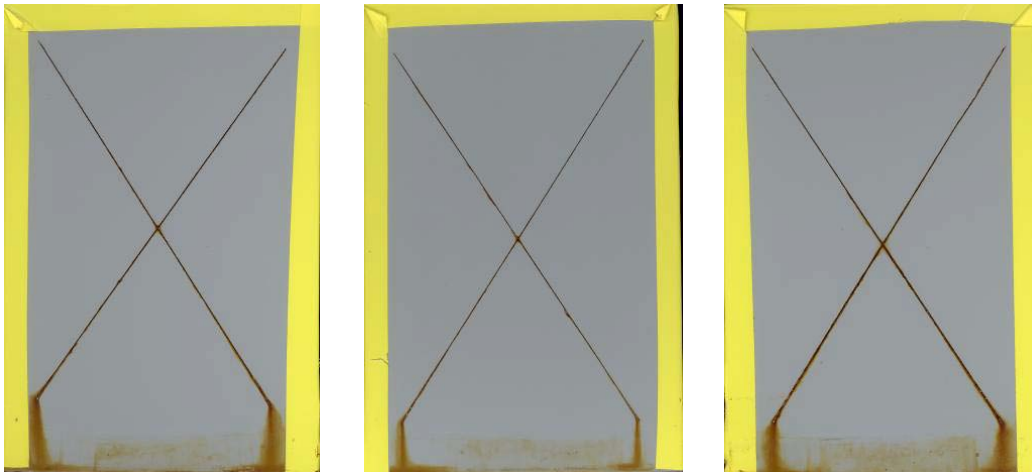
6.1.4.1.3 Cyclic SO₂ Salt Spray (Fog) Corrosion Resistance of Scribed Painted Coatings (HSS JTP Sec. 4.1.3)

In the painted and scribed acidic salt spray corrosion test the best protection from red rust at the scribes was provided by the aluminum coatings, as would be expected from the bare coating results presented earlier. Both cadmium and LHE Zn-Ni performed very similarly with each primer/paint system, whereby each experienced red rust formation early in the test which filled the scribe, although there was little or no blistering of the paint system adjacent to the scribe or in the field areas (Figures 60 and 61, respectively). For the aluminum coatings, there was little or no red rust observed throughout the entire duration of the test, however, significant field blistering and adhesion issues were noted as the test progressed.

The IVD-Al coated panels are shown in Figure 62. The panels primed with MIL-PRF-23377-C2 are shown at 500 hours (Fig. 62a); those primed with MIL-PRF-23377-N are shown at 668 hours (Fig. 62b) where they are holding up quite well. Those primed with 85582-N are shown at 840 hours (Fig. 62c) where there was significant blistering failure of the paint system. Plotted graphically with time, the IVD-Al panels consistently received the lowest ratings under each paint system as may be seen in Fig. 65a-c. The Alumiplaste™ coating results are shown in Figure 63 (1168 hours). With the MIL-PRF-23377-C2 primer, there was a small amount of blistering at the scribe. With the 23377-N primer, larger blisters are evident along the scribe (Fig. 63b), while the 85582-N primer also showed significant blistering along the scribes. The sputtered Al panels shown in Figure 64 exhibited blistering failures initiating from the bottom edge of the panels, which were not as well protected on the backsides or bottom edges as well as the other aluminum coatings. This is because both IVD and electroplating processes are capable of coating backsides/edges, whereas sputtering is a line-of-sight process. Nevertheless, it may be

observed that the 85582-N primer resisted blistering the least of the three primers in this study by comparing Fig. 64c with Fig. 64a-b. Graphically, the ratings for each primer/paint system are provided in Figure 65a, b, and c. Fairly consistent relative performance was observed for the cadmium alternative coatings in each primer/paint system indicating that the sacrificial coating is the dominant protector of the underlying steel surface.

Results of this accelerated corrosion test are influenced by the quality of the conversion coating applied by the coating vendors. In the case of the sputtered Al, the panels arrived at PAX for test in the non-conversion coated condition since the vendor did not have this capability. A fresh chromate conversion coating was applied the day prior to the primer application, so time-to-paint was not a factor for the observed paint adhesion failure on Sputtered Al. All panels were shipped by the vendors in a fairly tightly wrapped condition, and they were unpacked the day prior to primer application to minimize environmental degradation of the chromate conversion coatings. Figure 65 (a-c) contains graphs of the SO₂ corrosion resistance results for each primer tested.



(a) Painted, scribed cadmium panels with MIL-PRF-23377-C2 (1168 hr)

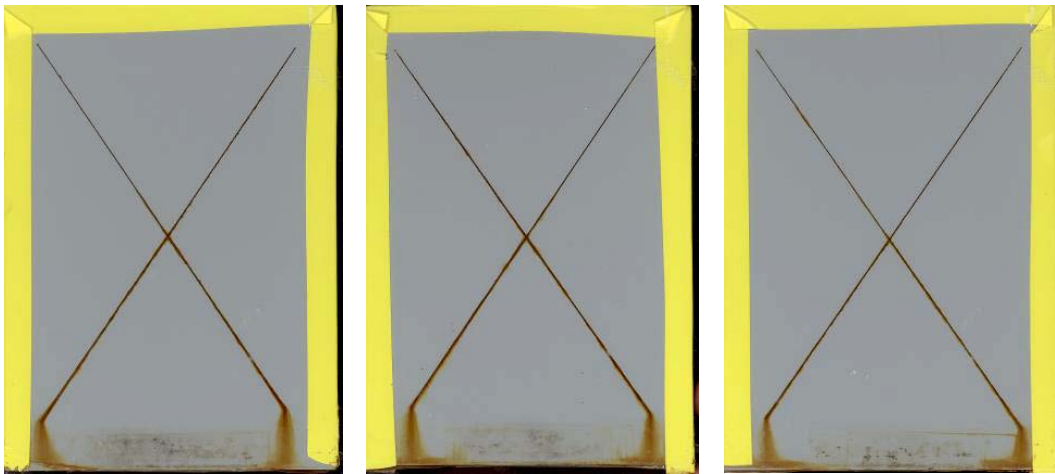


(b) Painted, scribed cadmium panels with MIL-PRF-23377-N (1168 hr)



(c) Painted, scribed cadmium panels with MIL-PRF-85582-N (1168 hr)

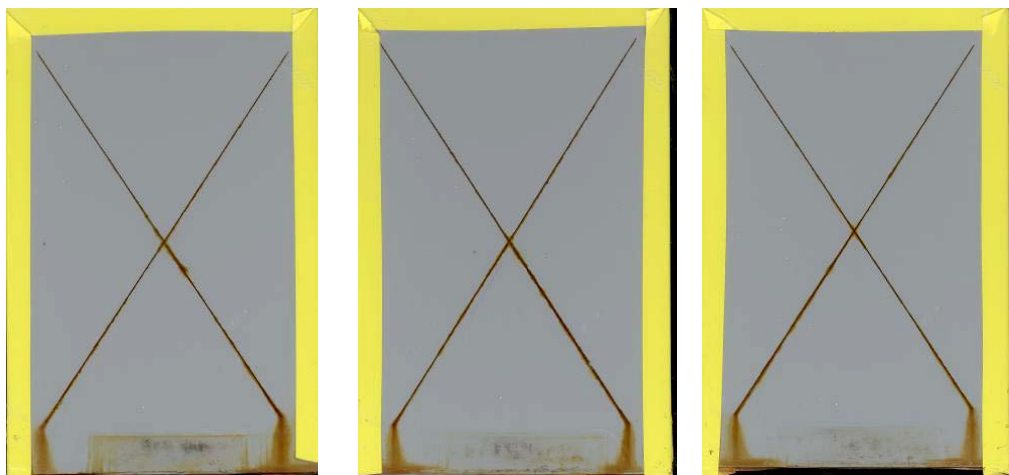
Figure 60. Scribed, Painted Cyclic SO₂ Salt Fog Corrosion Resistance (Cadmium)



(a) Painted, scribed panels with MIL-PRF-23377-C2 (1168 hr)

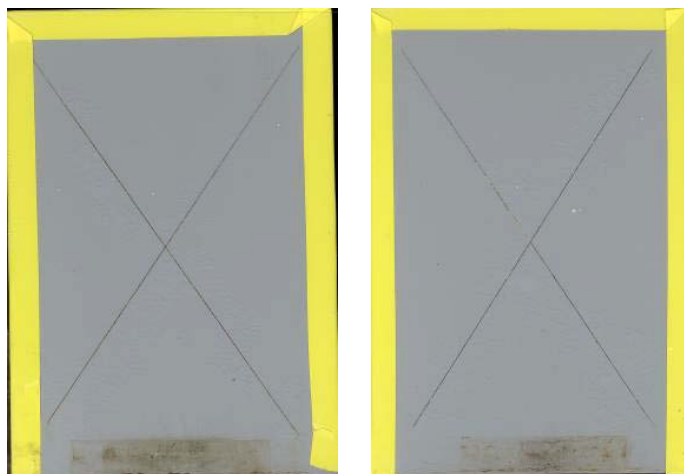


(b) Painted, scribed panels with MIL-PRF-23377-N (1168 hr)

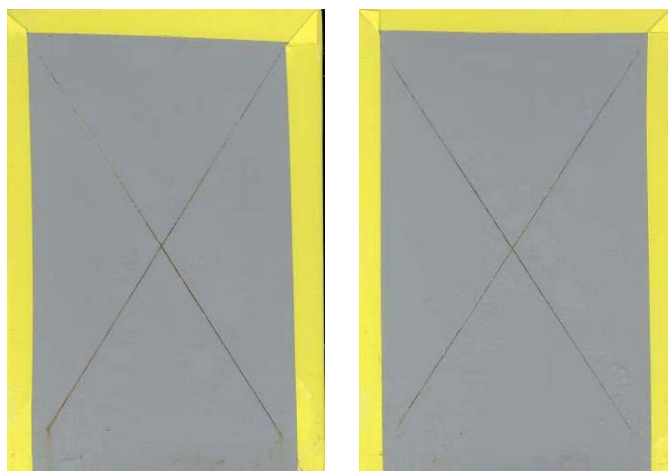


(c) Painted, scribed panels with MIL-PRF-85582-N (1168 hr)

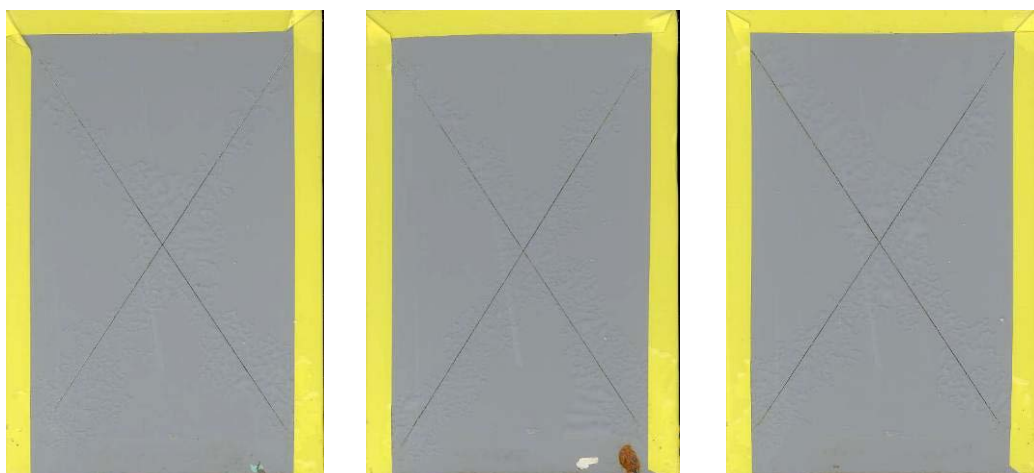
Figure 61. Scribed, Painted Cyclic SO₂ Salt Fog Corrosion Resistance (LHE Zn-Ni)



(a) Painted, scribed panels with MIL-PRF-23377-C2 (500 hr)



(b) Painted, scribed panels with MIL-PRF-23377-N (668 hr)



(c) Painted, scribed panels with MIL-PRF-85582-N (840 hr)

Figure 62. Scribed, Painted Cyclic SO₂ Salt Fog Corrosion Resistance (IVD-AI)



(a) Painted, scribed panels with MIL-PRF-23377-C2 (1168 hr)



(b) Painted, scribed panels with MIL-PRF-23377-N (1168 hr)



(c) Painted, scribed panels with MIL-PRF-85582-N (1168 hr)

Figure 63. Scribed, Painted Cyclic SO₂ Salt Fog Corrosion Resistance (AlumiPlate™)



(a) Painted, scribed panels with MIL-PRF-23377-C2 (1168 hr)

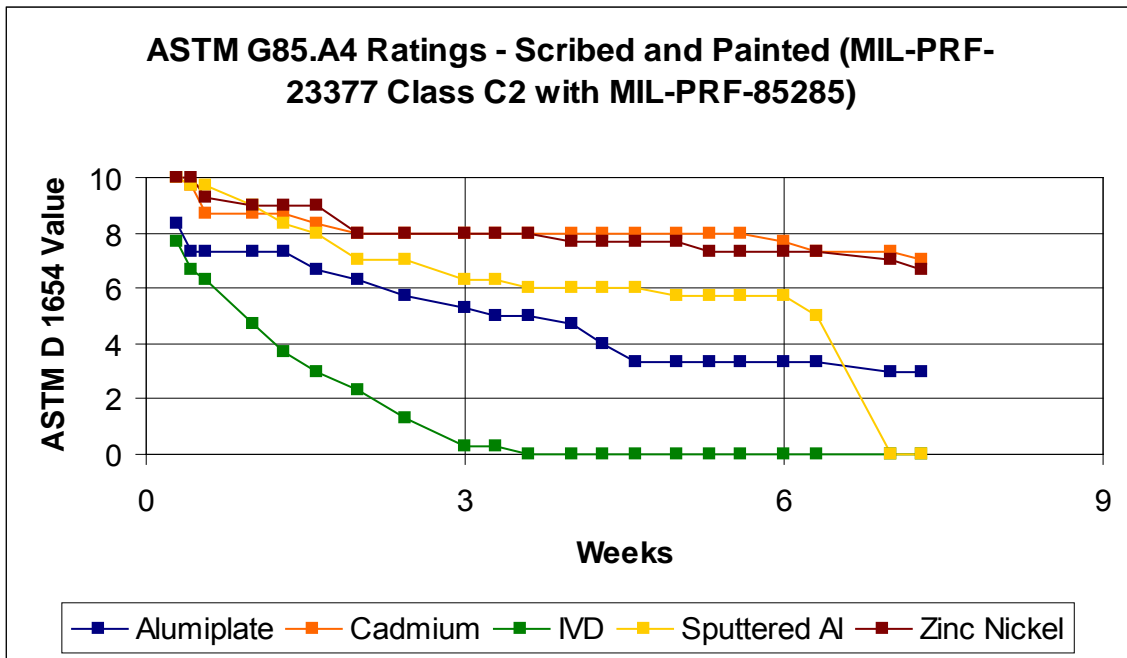


(b) Painted, scribed panels with MIL-PRF-23377-N (1168 hr)

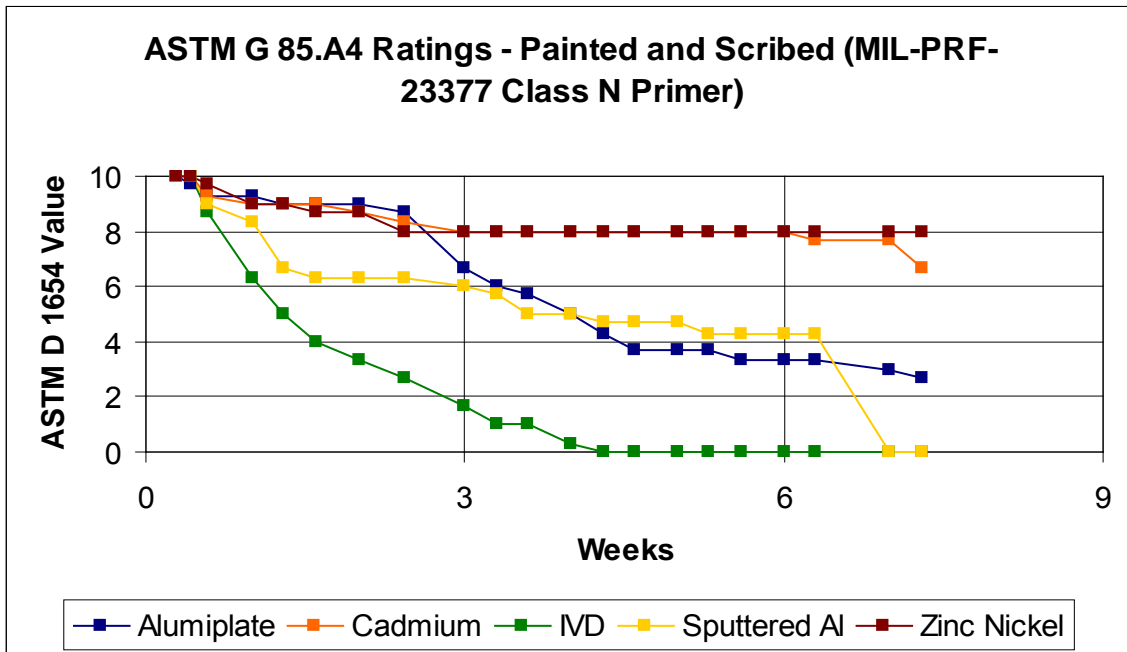


(c) Painted, scribed panels with MIL-PRF-85582 (1168 hr)

Figure 64. Scribed, Painted Cyclic SO_2 Salt Fog Corrosion Resistance (Sputtered Al)

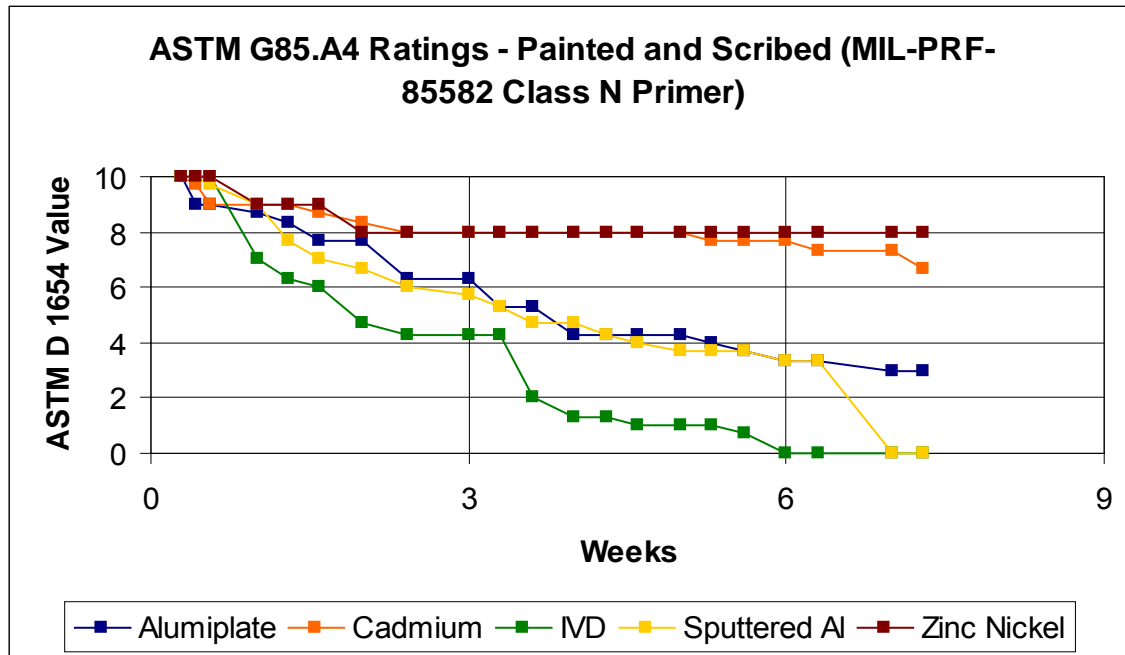


(a) ASTM G 85.A4 ratings for painted and scribed coatings using MIL-PRF-23377 Class C2 chromate inhibited primer



(b) ASTM G 85.A4 ratings for painted and scribed coatings using MIL-PRF-23377 Class N non-chromate inhibited primer

Figure 65. Scribed, Painted Cyclic SO₂ Salt Fog Corrosion Resistance (Ratings vs. Time)

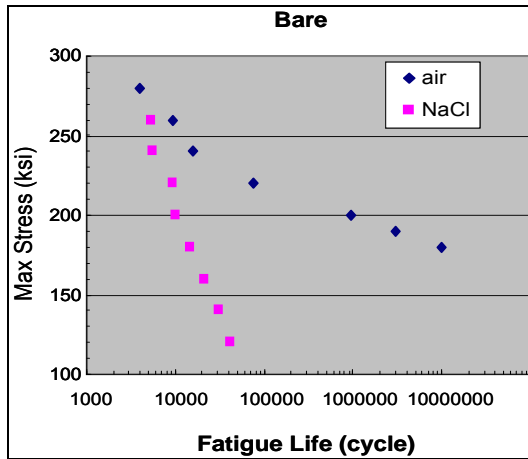


(c) ASTM G 85.A4 ratings for painted and scribed coatings using MIL-PRF-85582 Class N non-chromate inhibited primer

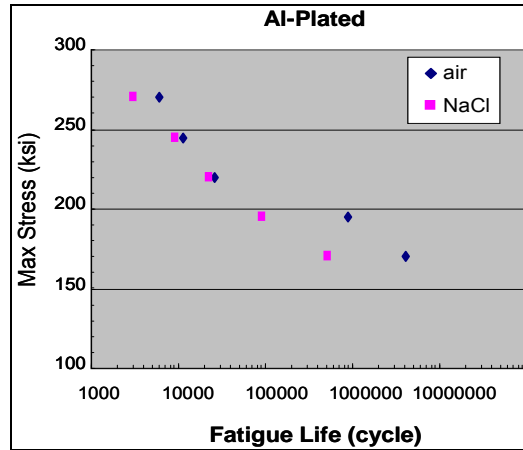
Figure 65 (cont'd). Scribed, Painted Cyclic SO₂ Salt Fog Corrosion Resistance (Ratings vs. Time)

6.1.4.1.4 Corrosion Fatigue Test (HSS JTP 4.1.4)

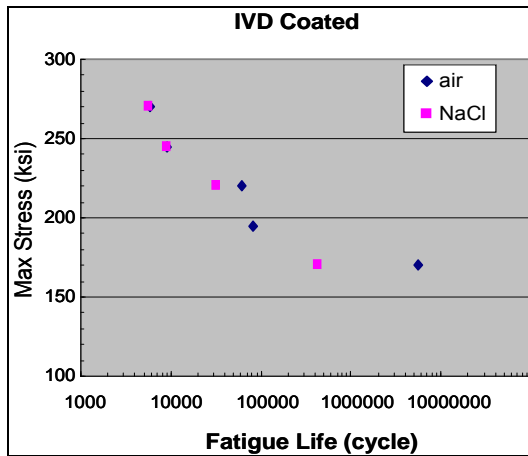
The stress-life (S-N) fatigue data for the bare and coated (unpainted) specimens, tested in air and 3.5% NaCl solution, are shown in Figure 66 where each plot represents a different cadmium alternative coating. Insufficient axial specimens were tested for structures engineers to draw statistically meaningful curves so this data was originally released in this same format at the 2007 Tri-Service Corrosion Conference [12]. However, enough data is available to see trends in performance among Cd, Zn-Ni coatings and the pure Al coatings. The fatigue life of the bare 4340 steel control specimen is, as expected, significantly shorter in 3.5% NaCl solution than in air, especially at lower applied stresses where corrosion-induced hydrogen embrittlement has more time to take place (Fig. 66(a)). On the other hand, the fatigue life of the coated specimens is only slightly reduced in 3.5% NaCl solution (NaCl data points in Figs. 66(b-f) are almost all slightly to the left of the air data points). The fatigue life data of all the bare and coated specimens were also co-mingled onto the plots shown in Fig. 67(a) (for air) and in Fig. 67(b) (3.5% NaCl environment). In air, the fatigue life of the coated specimens is generally shorter than that of the bare specimen. In 3.5% NaCl solution, the fatigue life of the coated specimens is longer than those of the bare specimen, Figure 67(b). This observation shows that all these sacrificial coatings generally improve corrosion fatigue resistance in 3.5% NaCl solution. Inspecting individual data points in Fig. 67, it appears that a slightly larger fatigue debit is produced by the Zn-Ni coatings under this fatigue test condition/geometry since they most frequently appear toward the left side of the plot.



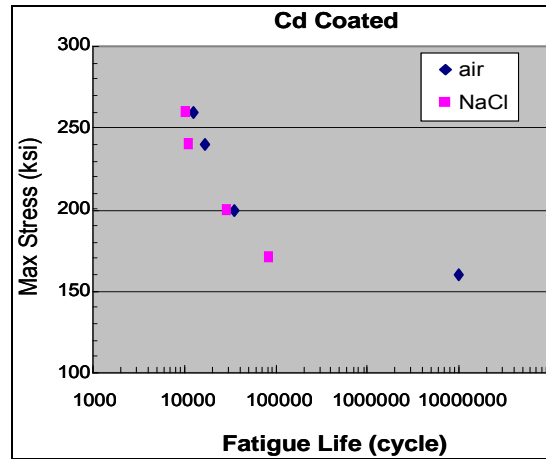
(a)



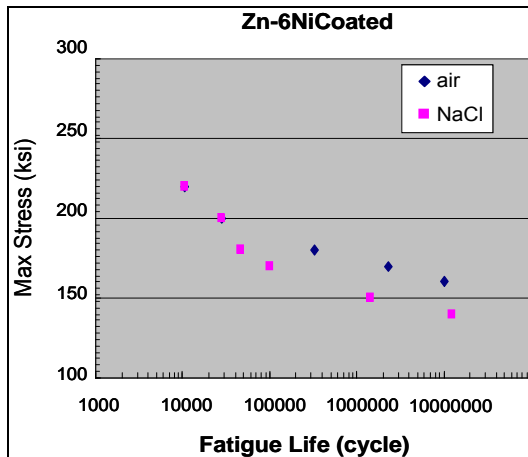
(b)



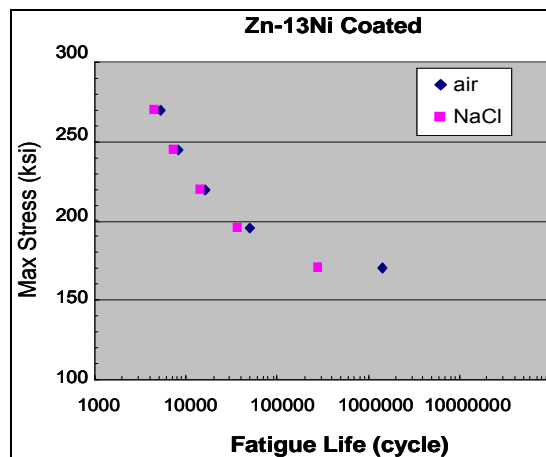
(c)



(d)

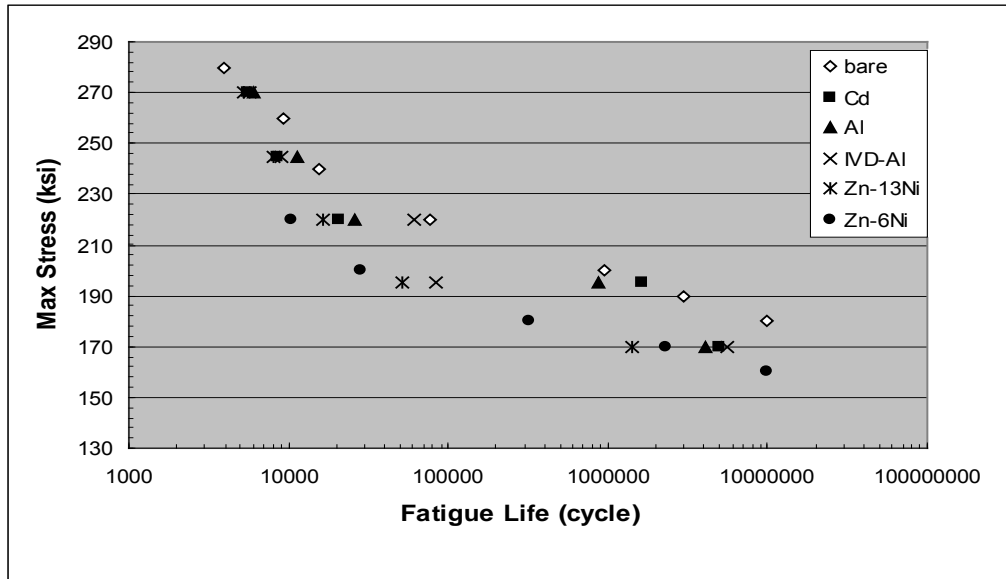


(e)

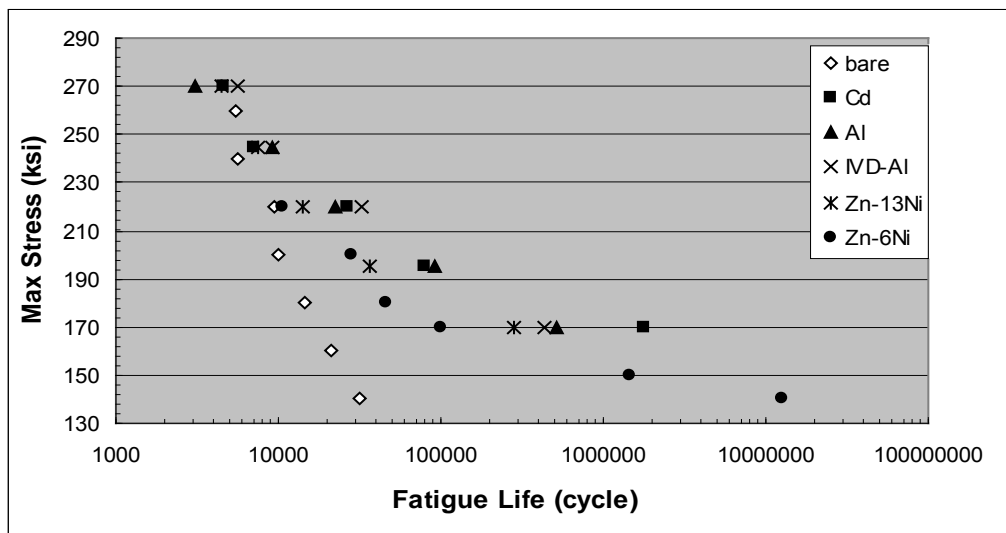


(f)

Figure 66. Stress Life Curves of (a) Bare, (b) AlumiPlate[™], (c) IVD Al, (d) Cd Coated, (e) Zn-6Ni, and (f) Zn-13Ni in Air and 3.5% NaCl Solution



(a) Air



(b) 3.5% NaCl

Figure 67. Comparison of Fatigue Lives of Bare and Plated Specimens in (a) Air and (b) 3.5% NaCl Solution

6.1.4.3 Stress Corrosion Cracking and Susceptibility to Hydrogen Embrittlement (HSS JTP Sec. 4.3)

The variations of K_{OSCC} and K_{ISCC} with applied electric potential V_{SCE} are shown for the bare 4340 steel control specimen in Figure 68. With increasing V_{SCE} , K_{OSCC} increases steadily, but K_{ISCC} exhibits the highest value at $V_{SCE} = -1$ volt and subsequently decreases. Their values at the OCP -0.64 volt are $K_{OSCC} = 98.5 \text{ MPa}\sqrt{\text{m}}$ (89.6 $\text{ksi}\sqrt{\text{in}}$) and $K_{ISCC} = 11.5 \text{ MPa}\sqrt{\text{m}}$ (10.5 $\text{ksi}\sqrt{\text{in}}$), respectively. This data confirm that the un-precracked specimen has a significantly greater resistance to SCC than the precracked specimen in the applied V_{SCE} potential range studied.

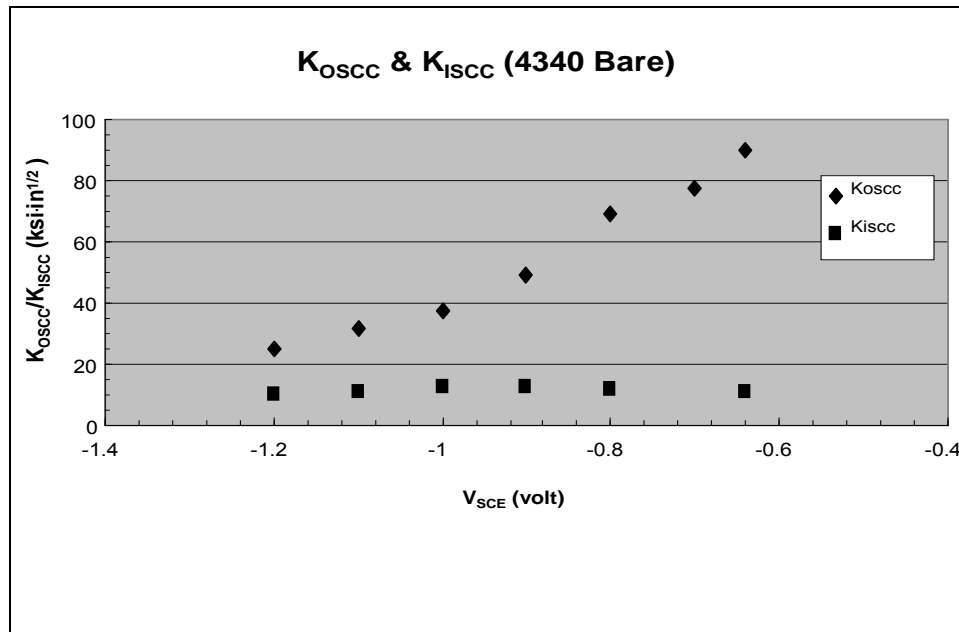


Figure 68. Variation of Threshold Stress Intensity for SCC in Un-Precracked (As-Machined) and Precracked Bare Specimens (K_{OSCC} and K_{ISCC} , respectively) with Applied Electric Potential V_{SCE}

The variation of K_{OSCC} with applied electric potential (V_{SCE}) for the coated specimens plotted along with the bare control specimen is shown in Figure 69. Within the region of V_{SCE} taken, the K_{OSCC} is greatest for the AlumiPlate™ coating compared to the other coatings, indicating it exhibits the greatest SCC resistance in 3.5% NaCl solution in this test. The values of open circuit potential (OCP) and the corresponding K_{OSCC} are listed for the bare and coated specimens in Table 73. Each Cd alternative coating had higher K_{OSCC} values than cadmium except for the Zn-6Ni coating.

It should be noted that the AlumiPlate™ coating thickness was substantially greater than any of the other coatings, and in particular LHE Cd which was the thinnest coating of the group (Table 73). Measurements were taken for residual stress on each of the alternative coatings and the results are also presented in Table 73. Most coatings had either slightly compressive or slightly

tensile stresses (<9 ksi) except for the Zn-6Ni coating which had a significant +46 ksi residual tensile stress. High residual tensile stress can lead to reduced substrate mechanical properties.

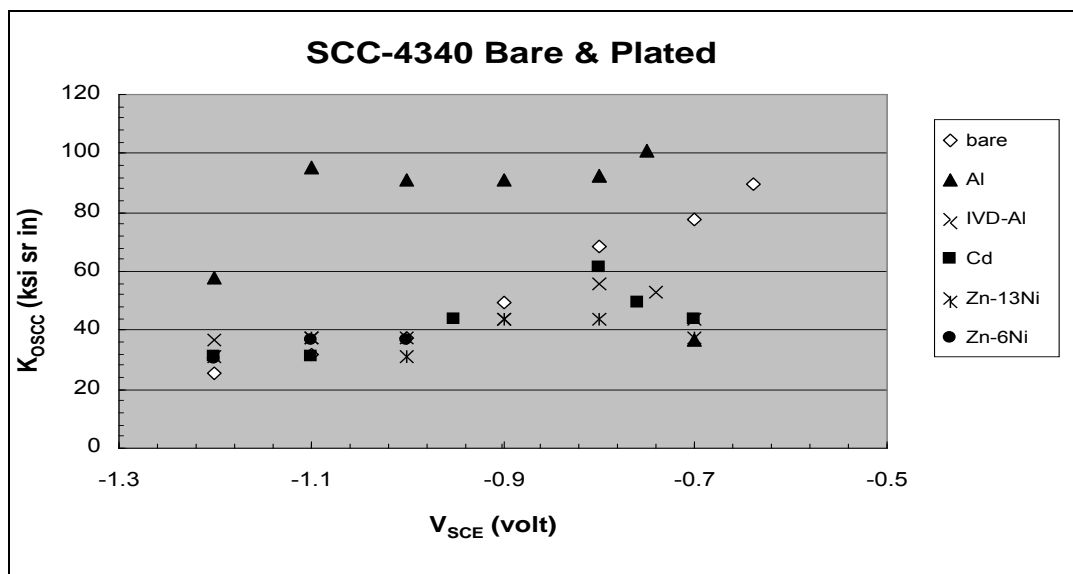


Figure 69. Variation of K_{OSCC} with V_{SCE} for Bare and Coated Specimens

Table 73: Open Circuit Potentials (OCP) and Threshold Stress Intensities for Stress Corrosion Cracking of Un-Precracked Specimen K_{OSCC} of Bare and Plated Specimens

Coating	OCP (Volt)	K_{OSCC} [MPa√M (KSI√IN)]	Coating Thickness, μm (mils)*	Coating Residual Stress [MPa (ksi)]*
Bare	-0.64	98.5 (89.6)	N/A	N/A
AlumiPlate™	-0.75	111.0 (101.0)	55 (2.2)	+21 (+3)
IVD-Al	-0.74	57.9 (52.7)	13 (0.5)	-61 (-8.8)
Cadmium	-0.76	54.4 (49.5)	9 (0.35)	-22 (-3.2)
Zn-6Ni	-1.00	40.4 (36.8)	16 (0.63)	+319 (+46.3)
Zn-13Ni	-0.75	61.8 (56.2)	12 (0.5)	-25 (-3.6)
* As measured by an independent laboratory used by NAVAIR				

6.1.5 U.S. Air Force Added Requirements (HSS JTP Sec. 5.0)

6.1.5.1 Acceptance criteria for Section 5.1.3.4.2 Torque Tension (HSS JTP Sec. 5.1)

The torque-tension versus load curves presented in Section 6.1.3.4.2 were different enough from the cadmium reference curves that it may be stated that no coating tested meets this requirement when using the single thread treatment selected for testing in this effort (anti-seize lubricant per AMS 2518). Considering that fasteners are often general use commodities and may be inserted and removed multiple times, this criterion is important to maintain required clamp up forces and loads on the structures into which they are installed. To implement alternative coatings where threaded features are present, additional effort would need to be directed toward appropriate thread treatments to minimize the scatter observed here.

6.1.6 U.S. Army Added Requirements (HSS JTP Sec. 6.0)

6.1.6.1 AMCOM In-Service Hydrogen Re-Embrittlement/SCC (HSS JTP Sec. 6.1)

AMCOM (U.S. Army Aviation and Missile Command) hydrogen re-embrittlement testing was conducted for all primary coatings. In this test ASTM F 519 Type 1d, 4340 high strength steel notched C-rings were self loaded to 65% NFS and placed into GM 9540P cyclic salt spray to determine whether the coating systems were resistant to in-service embrittlement. Nine replicates of each coating were tested. The acceptance criteria for this test were to be based on time to failure for the LHE Cd C-rings but were never formally established. This test was conducted at the U.S. Army Research Laboratory, and test results are shown in Table 74.

Essentially this test was too harsh to adequately discriminate performance among some of the coatings due to the short failure times that were observed (only a few hours). The data clearly show that AlumiPlate™ outperformed all coatings by a large margin, including LHE Cd. As stated earlier, the AlumiPlate™ coating is relatively more dense than the other coatings and its excellent performance could be due in part to it providing superior barrier properties. The AlumiPlate™ coated C-rings lasted an average 23.9 cycles, or 510 hours. Sputtered Al and LHE Cd had the next best performance, lasting an average of 396 minutes and 250 minutes, respectively. The remainder of the C-rings failed within 78 minutes of exposure. The Sn-Zn C-rings (later found to have not been properly embrittlement relieved) failed upon mechanical loading and prior to salt spray exposure.



Figure 70. Army C-ring Specimens in the Salt Spray Cabinet

Table 74. AMCOM Hydrogen Re-Embrittlement/Stress Corrosion Cracking Test Results

Specimen Number	Beginning Width	Final Width at Fracture	Hours Until Failure	Raw GM 9540P Minutes	**GM 9540P Cycles Completed	Relative Ranking
Bright 1	1.960	1.863	< 1	N/A	N/A	N/A - Sensitivity Test
Bright 2	1.962	1.865	< 1	N/A	N/A	
Bright 3	1.960	1.863	< 1	N/A	N/A	
Dull 1*	1.962	1.865	<4	N/A	N/A	
Dull 2*	1.962	1.865	<2	N/A	N/A	
Dull 3*	1.963	1.866	<23	N/A	N/A	
Dull 4*	1.965	1.868	No Failure	N/A	N/A	
Dull 5*	1.964	1.867	230 - 290	N/A	N/A	
Dull 6*	1.961	1.864	No Failure	N/A	N/A	
Plain 1	1.959	1.862	No Failure	N/A	N/A	
LHE Cd	1.958	1.874	N/A	163	0.1272	Control
LHE Cd	1.957	1.873	N/A	480	0.3747	
LHE Cd	1.960	1.876	N/A	129	0.1007	
LHE Cd	1.956	1.872	N/A	123	0.0960	
LHE Cd	1.959	1.875	N/A	176	0.1374	
LHE Cd	1.952	1.868	N/A	138	0.1077	
LHE Cd	1.958	1.874	N/A	480	0.3747	
LHE Cd	1.957	1.873	N/A	480	0.3747	
LHE Cd	1.953	1.869	N/A	85	0.0664	
LHE Cd, Average				250	0.1955	
IVD Al	1.962	1.878	N/A	38	0.0297	3
IVD Al	1.960	1.876	N/A	22	0.0172	
IVD Al	1.960	1.876	N/A	33	0.0258	
IVD Al	1.960	1.876	N/A	19	0.0148	
IVD Al	1.959	1.875	N/A	31	0.0242	
IVD Al	1.959	1.875	N/A	24	0.0187	
IVD Al	1.956	1.872	N/A	480	0.3747	
IVD Al	1.958	1.874	N/A	29	0.0226	
IVD Al	1.958	1.874	N/A	30	0.0234	
IVD Al, Average				78	0.0612	
Al Sputter	1.958	1.874	N/A	135	0.1054	2
Al Sputter	1.955	1.871	N/A	480	0.3747	
Al Sputter	1.960	1.876	N/A	480	0.3747	
Al Sputter	1.957	1.873	N/A	480	0.3747	
Al Sputter	1.959	1.875	N/A	480	0.3747	
Al Sputter	1.956	1.872	N/A	36	0.0281	
Al Sputter	1.956	1.872	N/A	480	0.3747	
Al Sputter	1.958	1.874	N/A	480	0.3747	
Al Sputter	1.952	1.868	N/A	511	0.3989	
Al Sputter, Average				396	0.3090	

Table 74 (cont'd). AMCOM Hydrogen Re-Embrittlement/SCC Test Results

Alumiplate	1.964	1.880	N/A	42273	33	1
Alumiplate	1.956	1.872	N/A	7686	6	
Alumiplate	1.959	1.875	N/A	38430	30	
Alumiplate	1.958	1.874	N/A	20	0.0156	
Alumiplate	1.956	1.872	N/A	46116	36	
Alumiplate	1.956	1.872	N/A	37149	29	
Alumiplate	1.957	1.873	N/A	47397	37	
Alumiplate	1.959	1.875	N/A	28	0.0219	
Alumiplate	1.953	1.869	N/A	56364	44	
Alumiplate, Average				30607	23.8931	
ZnNi Acid Boeing	1.958	1.874	N/A	25	0.0195	4
ZnNi Acid Boeing	1.957	1.873	N/A	38	0.0297	
ZnNi Acid Boeing	1.958	1.874	N/A	110	0.0859	
ZnNi Acid Boeing	1.953	1.869	N/A	50	0.0390	
ZnNi Acid Boeing	1.958	1.874	N/A	44	0.0343	
ZnNi Acid Boeing	1.958	1.874	N/A	35	0.0273	
ZnNi Acid Boeing	1.952	1.868	N/A	29	0.0226	
ZnNi Acid Boeing	1.959	1.875	N/A	190	0.1483	
ZnNi Acid Boeing	1.961	1.877	N/A	37	0.0289	
ZnNi Acid Boeing,				62	0.0484	
ZnNi Alk LHE D.G.	1.960	1.876	N/A	124	0.0968	5
ZnNi Alk LHE D.G.	1.961	1.877	N/A	35	0.0273	
ZnNi Alk LHE D.G.	1.960	1.876	N/A	45	0.0351	
ZnNi Alk LHE D.G.	1.957	1.873	N/A	57	0.0445	
ZnNi Alk LHE D.G.	1.958	1.874	N/A	27	0.0211	
ZnNi Alk LHE D.G.	1.957	1.873	N/A	24	0.0187	
ZnNi Alk LHE D.G.	1.957	1.873	N/A	40	0.0312	
ZnNi Alk LHE D.G.	1.957	1.873	N/A	40	0.0312	
ZnNi Alk LHE D.G.	1.958	1.874	N/A	33	0.0258	
ZnNi Alk LHE Dipsol, Average				47	0.0369	
SnZn (1st Batch)	1.960	1.876	<10min*	0	0	6
SnZn (1st Batch)	1.959	1.875	<10min*	0	0	
SnZn (1st Batch)	1.958	1.874	<10min*	0	0	
SnZn (1st Batch)	1.957	1.873	<10min*	0	0	
SnZn (1st Batch)	1.953	1.869	<10min*	0	0	
SnZn (1st Batch)	1.958	1.874	<10min*	0	0	
SnZn (1st Batch)	1.953	1.869	<10min*	0	0	
SnZn (1st Batch)	1.960	1.876	<10min*	0	0	
SnZn (1st Batch)	1.959	1.875	<10min*	0	0	
SnZn, Average				0	0.0000	
* C-rings failed before going into the salt spray chamber						
** 21.35 hours/cycle						

7.0 COST ASSESSMENT

As the objective of this project was head-to-head coatings assessment rather than demonstration of one particular coating technology, a detailed cost assessment was not prepared for each alternative tested here, however, a limited scope cost benefit analysis (CBA) was prepared by Concurrent Technologies Corporation through a leveraged effort with JG-PP. The Type B CBA which was contracted out is less detailed than a Type A analysis, but offers an approximation of cost impacts for potential coating process changes. Unfortunately, LHE Zn-Ni was not included in these calculations as the data was not available during preparation of the report. The analysis essentially compared electroplated aluminum with the vacuum based sputtered aluminum. The CBA results were generated using P2/FINANCE (Pollution Prevention Financial Analysis and Cost Evaluation System) software and government issued guidelines for determining return on investment (ROI). Two DoD facilities that were willing to respond to the surveys were used in the estimates: Navy FRC-E and Hill AFB. Detailed results of the CBA are presented in the report “Final Type B Cost Benefit Analysis of Cadmium Electroplating Alternatives”, Sept. 17, 2007 [30]. The referenced report is Distribution D due to proprietary process cost information provided by the participating coating vendors, however, the following general observations may be made.

Due to the high cost of the cadmium medical surveillance program (CMSP) at FRC-E, it was estimated that potential savings in the millions of dollars per year could exist for transitioning to what is determined by the Navy to be an equivalently performing cadmium alternative. Other DoD facilities may not have analogous medical surveillance programs based on the levels of cadmium exposure observed and will not have as strong of a cost driver. Neither sputtered aluminum nor AlumiPlate™ could function as drop-in replacements for cadmium in the varied applications at depots such as FRC-E and thus would reduce only a portion of the cadmium exposures, and legacy aircraft returning for rework will be carrying cadmium for many years. LHE Zn-Ni formulation(s) have the potential to replace the largest cadmium workload at DoD facilities and being essentially a drop-in replacement (due to the aqueous electroplating process) it is apparent that it would deliver a very competitive cost in applications where performance has been determined to be equivalent to cadmium. Based on performance in certain tests such as in-service embrittlement and fatigue it is not yet known if the coating can fully meet Navy performance requirements so a meaningful cost impact assessment is not possible.

8.0 IMPLEMENTATION ISSUES

Specific component engineering approval will be required to replace cadmium with any of the alternatives tested here due to at least some differences in key performance parameters—none fit across the board with regard to form, fit and function of cadmium (performance at similar thickness) in each JTP test. However, the two top performing cadmium alternatives according to the results of the Phase I and Phase II testing are AlumiPlate™ and LHE Zn-Ni while sputtered aluminum provides increased process flexibility to existing IVD-Al coated components. LHE Zn-Ni will provide the lowest cost alternative where it is determined to be technically acceptable.

AlumiPlate™ Coating Implementation

NAVAIR issued an authorization letter for AlumiPlate™ coating on 25 June 2009 which limits the coating to applications and finish systems specifically addressed within the letter. Based upon Navy field validation of IVD-Al using legacy chromate-inhibited epoxy primers and polyurethane topcoats the coating was authorized to be used in similar applications where the additional throwing power of the electroplated coating would offer additional in-service embrittlement protection of high-strength components. The coating is therefore not authorized for bulk unpainted applications like electrical connectors and fasteners. The process is currently sole-source and proprietary so that parts must be sent to the vendor for coating which makes OEM level implementation more feasible than DoD Depot level. Also, the AlumiPlate™ process includes the use of toluene and a hazardous organometallic electrolyte. So, although it does eliminate the use of cadmium (and potentially Cr⁺⁶), the process itself still uses hazardous chemicals. The AlumiPlate™ authorization letter represents only one facet in the cadmium replacement effort and does not resolve the full scope problem of cadmium and hexavalent chromium (post-treatments and primers). There still remains a need to find appropriate cadmium alternatives for uses such as electrical connectors and fasteners beyond the scope of this project.

The following is a list of reported field testing or implementation of AlumiPlate™ on high strength steel substrates relevant to this project.

- CH-53K Super Stallion – HSS landing gear
- F-35 – Field test adjacent to cadmium part on HSS landing gear
- AH-1 Super Cobra – M50 HSS rotor hub housing
- C-5 Galaxy – Aircraft wheel fuse plugs
- DDG-1000 – HSS structural applications, deck hatch hinges
- Howitzer – HSS eye bolts field tested in Hawaii

LHE Zn-Ni (IZ-C17) Coating Implementation

Scale-up efforts for LHE Zn-Ni process optimization are being performed by Hill AFB in conjunction with contractors and partners. The extent of actual implementation remains to be seen. NAVAIR will be conducting further Navy-specific testing and optimization efforts in FY11-12 at FRC-SE through a NESDI funded effort.

In addition to the further work just mentioned, Dipsol of America recently optimized their LHE zinc-nickel formulation for extended bath lives for high Amp-hour production schedules (identified as IZ-C17+). The US Air Force in conjunction with its contractor team has scaled-up the optimized LHE Zn-Ni+ plating for potential landing gear applications. Component designs are being reviewed to identify suitable applications. NAVAIR would like to evaluate this new LHE Zn-Ni process for drop-in applications at FRC-SE and FRC-E with non-hexavalent chromium post-treatments, followed by carefully selected field demonstrations.

Magnetron Sputtered Aluminum Coating Implementation

Magnetron sputtered aluminum coatings had been implemented at HAFB/OO-ALC during or prior to the course of this project. As discussed earlier the sputter capability is intended to be augmented to existing IVD-Al vacuum chambers making it a n incremental improvement requiring less capital than stand-alone sputter coating equipment. Limited applications are currently authorized so the equipment experiences intermittent operation. Specific applications by platform are not authorized for inclusion here.

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APPENDIX A: POINTS OF CONTACT

Points of contact who were most recently involved with ESTCP Project WP-0022 are listed below.

POINT OF CONTACT Name	ORGANIZATION	Phone/Fax/e-mail	Role in Project
Beck, Erin	NAVAIR 4.3.4.2	(301) 342-6183	NAVAIR P.I. Phase I
Berman, Elizabeth	AFRL	(937) 656-5700	AFRL P.I. Phase II
Brown, Steven	NAVAIR 4.3.4.2	(301) 342-8101	NAVAIR P.I. Phase II
Debias, Leanne	Concurrent Technologies Corporation	(814) 269-6830	JTP Execution Coordinator
Donaldson, Kelly	AlumiPlate, Inc.	(763) 786-3788	Coating vendor
Fowler, Amy	NAVAIR 4.3.4.2	(301) 342-0986	Project lead for NESDI funded Navy Specific testing
Gaydos, Stephen	Boeing (St. Louis)	(314) 233-3451	OEM Representative
Hughes, Nathan	Hill AFB	(801) 775-2270 Nathan.hughes@hill.af.mil	USAF Depot
Kunihiro, Takeshi	Dipsol of America	(734) 261-0633	Coating vendor
Lee, Eun	NAVAIR 4.3.4.1	(301) 342-8069	SCC and Fatigue Investigator
Marshall, John	Marshall Laboratories	(303) 442-0156	Coating vendor
Wass, Lorraine	Joint Group on Pollution Prevention	(207) 384-5249	NAVAIR Contractor, JG-PP WG POC

APPENDIX B: COATING QUALITY AND PROCESS INFORMATION

Coating thickness was measured by NAS Pax River on 4x6" flat steel panels using an Elcometer 456 Coating Thickness Gauge with ferrous F1 probe. Accuracy of the gauge was ± 0.1 mil.

Coating: Sputtered Aluminum

Coater: Marshall Laboratories

Processing Sequence (*all specimen types*):

- Grit blast
- Isopropyl ultrasonic rinse
- Glow discharge clean for 15 minutes in 20mTorr argon atmosphere.
- Plug and coat sputter process. Specimens were mounted radially around the cathode approximately 4" away from cathode surface. Specimens were sputtered for 2.5 hours at about 8 kW power, except for the 1a.1 round bar specimens which used special parameters to limit heat input to these small specimens (8 hours at about 1.6 kW). Targeting coating thickness was 0.6 to 0.8 mils.

Conversion coating was applied to flat panels and round bars at NAS Patuxent River Inorganic Coating Laboratory according to the process below (except for the paint adhesion test flat panels, conversion coated at CTC).

- Deionized water rinse
- Conversion coat in Alodine 1200S for 90-120 seconds at room temperature
- Tap water rinse
- Deionized water rinse
- Air dry

Coating composition: 100% aluminum

Coating thickness: 0.6 to 0.8 mils targeted.

Coating coverage: Type 1a.1 specimens were inspected by NAS Pax River for coverage in the notch using 40X magnification prior to HE tests. All specimens appeared to have full coating coverage in the notch. There were light grey spots on four specimens. One specimen had a small scratch on the notch skirt. Type 1d specimens were inspected by NAS Pax River for coverage in the notch using 3.5X magnification. All specimens appeared to have full coating coverage in the notch. Flat panel specimens prepared for Phase II testing were not coated on both sides which led to reduced quality of results in comparison to the other aluminum coatings.

Coating: Ion Vapor Deposited (IVD) Aluminum (unpeened)

Coater: Hill Air Force Base

Processing Sequence (*Type 1a.1 notched round bars*):

- Degrease with acetone
- Fixture
- Degrease with acetone
- Mask threads
- Abrasive blast using 100 grit garnet at 50 psi with the nozzle 4 to 6 inches from the part.
- All specimens plated together in one batch with a production run in a production coater. Time between blast and application of vacuum in the coater was approximately 2 hours.

- Cool to room temperature
- Conversion coat (chromate)
- Rinse in cold running water
- Air blow dry

Coating composition: 100% aluminum

Coating thickness: Class 2 minimum (0.5 mil) targeted. Not measured.

Processing Sequence (1"x4" bend adhesion panels):

- Degrease with acetone
- Abrasive blast using 100 grit garnet at 50 psi with nozzle held about 6 inches from the panels.
- All panels were coated in one batch with a production run in a production coater. Time between blast and application of vacuum in the coater was approximately 3 hours.
- Conversion coat (chromate)
- Cold water rinse
- Air blow dry

Coating composition: 100% aluminum

Coating thickness: Class 2 minimum (0.5 mil) targeted. Not measured.

Coating coverage: Notch examined by coater under 10X magnification prior to conversion coating step – coverage appeared complete. Notch examined by coater under 10X magnification for full coverage. Four specimens showed full coverage into the root of the notch indicated by uniform conversion coating color. Six specimens showed non-uniform color in the root of the notch leading to questions about the notch coverage. Specimens re-examined at 30X. Believe the notch to be completely coated as evidenced by the lack of any corrosion in the notch and the examination prior to conversion coat. These six specimens were identified by a question mark on each bag in which they were stored. Specimens were inspected by NAS Pax River for coverage in the notch using 3.5X magnification. All specimens appeared to have full coating coverage in the notch.

Coating: Low Hydrogen Embrittlement (LHE) Cadmium

Coater: Hill Air Force Base

Processing Sequence (Type 1a.1 notched round bars):

- Degrease with acetone
- Fixture
- Degrease with acetone
- Mask threads
- Abrasive blast using 100 grit garnet at 40 psi with the nozzle 4 to 6 inches from the part.
- Plate per MIL-STD-870. Plated in production bath at 60 A/ft² for 8.0 minutes. Time from abrasive blast to plate approximately 10 minutes. Specimens were gang plated in three groups of six. The plating solution was continuously filtered and the parts were moved back and forth in the solution to give additional solution circulation around the parts.
- Cold water rinse
- Air blow dry
- Bake for 23 hours at 375°F. Time from plate to bake as follows: 1 hour 50 minutes for Batch 1, 1 hour for Batch 2 and 20 minutes for Batch 3.

- Activate in 1% sulfuric acid for 5 seconds
- Conversion coat (chromate)
- Running cold water rinse
- Air blow dry

Coating composition: 100% cadmium

Coating thickness: No attempt was made to measure coating thickness. Using cathode efficiency of 75% (which is typical of this solution at the current density used) 8.0 minutes will result in a coating weight equivalent to 0.6mil thick. Due to the porosity of the deposit, the actual deposit may be thicker than this.

Coating coverage: Notch examined by coater under 10X magnification for full coverage. Two of the 18 specimens showed spots that appeared bare (no cadmium coating). Those two specimens were identified by the coater with blue paper over the tissue wrap. Those two specimens were not used for testing. Specimens were inspected by NAS Pax River for coverage in the notch using 100X magnification after HE tests conducted. In general specimens appeared to have full coating coverage in the notch, on some specimens the coverage appeared slightly light to bare.

Processing Sequence (Type 1d notched C-rings):

- Degrease with acetone
- Abrasive blast using 100 grit garnet at 40 psi with the nozzle 4 to 6 inches from the part.
- Fixture
- Plate per MIL-STD-870. Plated in production bath. Plated inner diameter at 60 A/ft² for 8.0 minutes. Plated outer diameter at 57 A/ft² for 9.0 minutes. Time between blast and start of plate was approximately 30 minutes for all specimens. Specimens were plated together in one batch. The plating solution was continuously filtered and the parts were moved back and forth in the solution to give additional solution circulation around the parts.
- Cold water rinse
- Air blow dry
- Bake for 23 hours at 375°F. Time from plate to bake was 1 hour.
- Activate in 1% sulfuric acid for 5 seconds
- Conversion coat (chromate)
- Running cold water rinse
- Air blow dry

Coating composition: 100% cadmium

Coating thickness: 0.51 mils (0.10 standard deviation) measured by NAS Pax River (Type 1d specimens only).

Coating coverage: Notch examined by coater under 10X magnification prior to bake step – all specimens appeared to have full coverage in the notch. Notch examined by coater under 10X magnification after conversion coating step for full coverage. All specimens showed non-uniform color in the root of the notch leading to questions about the notch coverage. Specimens re-examined under 30X magnification. Believe the notch to be completely coated as evidenced by the lack of any corrosion in the notch and the examination prior to conversion coat. Specimens were inspected by NAS Pax River for coverage in the notch using 3.5X magnification. All specimens appeared to have full coating coverage in the notch.

Processing Sequence (1"x4" bend adhesion panels):

- Degrease with acetone
- Fixture (all panels were fixtured adjacent to each other lengthwise with no gaps between panels in an effort to reduce edge effects; panels were taped together across the top and bottom)
- Abrasive blast using 100 grit garnet at 40 psi with the nozzle held about 6 inches from the panels.
- Plate per MIL-STD-870. Plated in production bath at 60 A/ft² for 8.0 minutes. Time between blast and start of plate was approximately 10 to 15 minutes.
- Rinse
- De-mask
- Conversion coat (chromate)
- Cold water rinse
- Air blow dry

Coating composition: 100% cadmium

Coating thickness: No attempt was made to measure coating thickness. Eight minutes plating time is equivalent to 0.6 mil average thickness. However, it appeared that there was considerable edge effect likely resulting in heavier deposit around the outside edges.

Coating: Electroplated Aluminum (AlumiPlate™)

Coater: AlumiPlate, Inc.

Processing Sequence (all specimen types):

- Grit blast with 80 grit Corundum
- Activate:
 - Oakite NST for 5 min. at 140°F ± 5°F
 - Dip rinse and dip-spray
 - Electroclean 1751 Anodic for 2 min. ± 5 sec. at 150°F ± 5°F
 - Dip rinse and dip-spray
 - Metex M-629 for 15 min. at ambient temperature
 - Dip rinse and dip-spray
 - Ammonia dip for 12 sec. at ambient temperature
 - Dip rinse and dip-spray
- Activate:
 - Inert activation for 15 sec. at 125°F
 - Rinse 7X
 - Toluene rinse 3X
- Plate per MIL-DTL-83488D Class 2, Type II, Class 1A (MIL-C-5541 chromate treatment).
- Rinse
 - DI spray rinse for 30 sec.
 - Nitric acid dip rinse for 15±5sec. at ambient temperature
 - DI spray rinse for 30 sec.
 - Dip rinse for 30 sec.
- Conversion coat in Iridite 14-2 for 1 min. 30 sec., Class 1A
- Dead rinse 2X
- DI spray/hose rinse

- Air dry

Coating composition: 100% aluminum (no underplate except on titanium alloy panels where a nickel strike was used)

Coating thickness: Class 2 (≥ 0.5 mil) certified. 0.92 mils targeted.

Coating coverage: Type 1a.1 specimens were inspected by NAS Pax River for coverage in the notch using 100X magnification after HE tests conducted. All specimens appeared to have full coating coverage in the notch. Type 1d specimens were inspected by NAS Pax River for coverage in the notch using 3.5X magnification. All specimens appeared to have full coating coverage in the notch.

Coating: Tin-Zinc Plating

Coater: Dipsol of America, Incorporated

Processing Sequence (Type 1a.1 notched round bars, 1"x4" bend adhesion panels):

- Soak Clean in strong alkaline degreaser (145SC) for 10 minutes at 50°C.
- Rinse
- Electro Clean in strong alkaline cleaner (331ECX) with anodic current for 5 minutes at 50°C.
- Rinse
- Tin-Zinc Plating in tin-zinc plating solution (SZ-240) at pH 6 to 7 for 19 minutes at 25°C and 2 A/dm² current density.
- Rinse
- Dry
- Bake for 15 hours at 240°F.
- Rinse
- Conversion coat in strong acid chromate for tin-zinc plating (SZ-248) for 40 seconds at 30°C.
- Rinse
- Dry

Coating composition: 75 to 85% tin and 15 to 25% zinc (per coater documentation).

Coating thickness: 0.4 to 0.5mil (per coater documentation), not measured by NAS Pax River lab.

Coating coverage: Type 1a.1 specimens were not inspected by NAS Pax River for coverage in the notch prior to HE tests. Eight extra, untested specimens were inspected for coverage in the notch using 100X magnification. All eight specimens appeared to have full coating coverage in the notch, however, the coating surface contained numerous blisters/pits and areas of red rust.

Coating: Zinc-Nickel, Boeing Acidic

Coater: Boeing Seattle

Processing Sequence (all specimen types):

- Clean
- Abrasive clean with 80 grit aluminum oxide
- Rinse
- Dilute hydrochloric acid pickle for 30 seconds
- Rinse
- Zinc-Nickel plate per BAC 5637
- Rinse

- Blow dry with compressed air
- Bake at 375°F for 23 hours
- Immersion in zinc nickel activator
- Rinse
- Chromate conversion coat
- Rinse
- Blow dry with compressed air

Coating composition: 90.5 to 91.5% zinc, 8.5 to 9.5% nickel

Coating thickness: 0.5 mil targeted. 0.29 mils (0.06 standard deviation) measured by NAS Pax (Type 1d specimens only).

Coating coverage: Type 1a.1 specimens were inspected by NAS Pax River for coverage in the notch using 100X magnification after HE tests conducted. All specimens appeared to have full coating coverage in the notch. One specimen that was tested in the 200-hr HE test had a small chip in the coating on the notch skirt. Type 1d specimens were inspected by NAS Pax River for coverage in the notch using 3.5X magnification. All specimens appeared to have full coating coverage in the notch.

Coating: Low Hydrogen Embrittlement Zinc-Nickel, Dipsol IZ-C17

Coater: Boeing St. Louis

Processing Sequence (all specimen types):

- TCE Vapor Degrease
- Abrasive clean with 180 – 220 grit aluminum oxide
- Rinse
- Apply IZ-C17 Alkaline Zinc-Nickel plate: 3 A/dm² – RT – 45 minutes
- Rinse
- Dry
- Embrittlement Relief Bake at 375 +/- 25°F for 24 hours
- Rinse
- Activate in F-0529 (tartaric acid): 2 ml/L – RT – 15 seconds
- Chromate conversion coat: IZ-258 @ 60 °C – 60 seconds
- Rinse
- Dry @ 60 °C – 10 minutes

Coating composition: 11.2 to 16.1% nickel per XRF measurements

Coating thickness: 0.5 mils targeted. 0.53 mils (0.11 standard deviation) measured NAS Pax River (Type 1d only).

Coating coverage: Coater plated the reduced gauge section of Type 1a.1 (no plating on threaded ends) – Visual inspection of notch showed that entire notch was plated. Type 1a.1 specimens were inspected by NAS Pax River for coverage in the notch using 40X magnification prior to HE tests. All specimens appeared to have full coating coverage in the notch. Type 1d specimens were inspected by NAS Pax River for coverage in the notch using 3.5X magnification. All specimens appeared to have full coating coverage in the notch.

Coating: Brush Cadmium Plating, SIFCO 2023

Coater: Boeing St. Louis

Processing Sequence (Type 1a.1 notched round bars, 1"x4" bend adhesion panels):

- Brush plating was performed with a carbon anode with cotton batting and polyester sleeving.
- Degrease with MPK
- Abrasive grit blast with 220 grit aluminum oxide.
- DI water rinse and immediately plate.
- Brush plating tool dipped in SIFCO 2023 Cd solution and power supply set at 4 volts.
- Brush plating tool applied to part to begin plating immediately at 4 volts and voltage was quickly raised to 6.5 to 7 volts while observing plating to make sure that burning (dark plating) was not occurring.
- Brush plating continued until correct amp-hrs for test specimen was obtained in order to achieve 0.5 mil minimum thickness.
- SIFCO 2023 plated very easily and quickly. No burning occurred during the plating process. However, plating was rough – especially at top edge of notch (for Type 1a.1 specimens) and this pulled threads out of the cover.
- Rinse in water.
- Immerse in SIFCO 5005 conversion coating solution for 15 seconds to develop yellow-brown color.
- Rinse
- Blow dry

Coating composition: 100% cadmium

Coating thickness: 0.5 mil targeted. Measurements conducted in Phase II by CTC indicated the coating averaged 1.34 mils.

Coating coverage: Per the coater, cadmium plating appeared to be uniform and the notch on the Type 1a.1 specimens appeared to also have good plating coverage, except for #AL4068 (had some flaking in the notch most likely due to poor surface prep). Type 1a.1 specimens were inspected by NAS Pax River for coverage in the notch using 40X magnification after HE tests were conducted. All specimens appeared to have full coating coverage in the notch. Untested specimens were also inspected at 40X magnification. Two of four untested specimens had exposed (uncoated) area in the notch root and the coating looked thin around the entire notch root of one specimen.

Coater notation: Type 1a.1 specimens were inspected and cloth threads were seen on the plating. These were cleaned with scotch brite pads and then conversion coated for another 15 seconds.

Coating: Brush Zinc-Nickel Plating, SIFCO 4018

Coater: Boeing St. Louis

Processing Sequence (Type 1a.1 notched round bars, 1"x4" bend adhesion panels):

- Brush plating was performed with a carbon anode with cotton batting and polyester sleeving.
- Degrease with MPK
- Abrasive grit blast with 220 grit aluminum oxide.
- DI water rinse and immediately plate.
- Brush plating tool dipped in SIFCO 4018 Zn-Ni solution and power supply set at 4 volts.

- Brush plating tool applied to part to begin plating immediately at 4 volts and voltage was quickly raised to 7 volts while observing plating to make sure that burning (dark plating) was not occurring.
- Brush plating continued until correct amp-hrs for test specimen was obtained in order to achieve 0.5 mil minimum thickness.
- SIFCO 4018 plated very easily but not as fast as LDC 5030. No burning occurred during the plating process.
- Rinse in water.
- Immerse in SIFCO 5030 conversion coating solution. No conversion coating formed at solution temperatures <110°F for the 1x4 panels and <130°F for the Type 1a.1 specimens. SIFCO 5030 had to be heated to 110° or 130°F in order to develop a brown conversion coating color. 30 seconds appeared to be sufficient at the elevated temperatures.
- Rinse
- Blow dry

Coating composition: 8 to 12% nickel, balance zinc (per SIFCO)

Coating thickness: 0.5 mil targeted. Measurements conducted in Phase II by CTC indicated the coating averaged 0.89 mils.

Coating coverage: Per the coater, Zn-Ni plating did not have a uniform appearance on some (8 of 18) of the notches on the Type 1a.1 specimens and are noted in documentation that accompanied the test specimens. The remainder of the specimens (10 of 18) appeared to have good plating coverage. Type 1a.1 specimens were inspected by NAS Pax River for coverage in the notch using 40X magnification prior to HE tests. All specimens except one had a shiny notch root possibly indicating thin coating. Three specimens appeared to have bare spots in the notch root. Only the specimens indicated as having good coating coverage by the coater were used for testing.

Coating: Brush Tin-Zinc Plating, LDC 5030

Coater: Boeing St. Louis

Processing Sequence (Type 1a.1 notched round bars, 1"x4" bend adhesion panels):

- Brush plating was performed with a carbon anode with cotton batting and polyester sleeving.
- Degrease with MPK
- Abrasive grit blast with 220 grit aluminum oxide.
- DI water rinse and immediately plate.
- Brush plating tool dipped in LDC 5030 Sn-Zn solution and power supply set at 4 volts.
- Brush plating tool applied to part to begin plating immediately at 4 volts and voltage was quickly raised to 10.5 or 15 volts while observing plating to make sure that burning (dark plating) was not occurring.
- Brush plating continued until correct amp-hrs for test specimen was obtained in order to achieve 0.5 mil minimum thickness.
- LDC 5030 plated very easily and quickly. No burning occurred during the plating process.
- Rinse in water.
- Immerse in LDC 1720 conversion coating solution for 3 to 10 seconds to develop yellow-brown color.
- Rinse

- Blow dry

Coating composition: 70 to 75% tin, balance zinc (per LDC)

Coating thickness: 0.5 mil targeted. Measurements conducted in Phase II by CTC indicated this target was met.

Coating coverage: Per the coater, Sn-Zn plating appeared to be uniform and the notch on the Type 1a.1 specimens appeared to have good plating coverage.

Coating: Spray-on SermeTel® 249/273

Coater: Boeing St. Louis

Processing Sequence (Type 1a.1 notched round bars, 1"x4" bend adhesion panels):

- SermeTel® 249/273 were applied per Sermatech Surface Engineering Bulletin 249 with the following exceptions.
 - Solvent clean test specimens with TCE vapor degreasing
 - Abrasive grit blast with 80 grit aluminum oxide.
 - Apply SermeTel® 249 at 1.0 to 2.0 mil coating thickness.
 - Dry (30 minutes)
 - Cure - brush apply SermeTel® 273 curing agent onto the 249 coating (curing method #2 in Sermatech Bulletin).

Coating composition: The coating contains aluminum and zinc powder in an inorganic binder system (per Sermatech)

Coating thickness: 1.0 to 2.0 mil targeted. Measurements conducted in Phase II by CTC indicated the coating averaged 1.42 mils.

APPENDIX C

This Appendix includes the bend specimen analysis report generated for Phase I of this project.

Cadmium Alternatives Sample Substrate Examination

B. Pregger NAVAIR 4.9.7.1

23 November 2005

Introduction

Two samples were examined with the scanning electron microscope (SEM) as a continuation of support for the cadmium alternatives hydrogen embrittlement and re-embrittlement tests. Examinations consisted of elemental analysis of the substrate exposed during coating adhesion testing. Specific information requested was whether the substrate exposed by coating spallation consisted of aluminum or steel.

The sample material was 4130 steel coated by (1) IVD aluminum, and (2) Alumiplate coating. The spalling surfaces were examined at high magnification using a JEOL 6460LV scanning electron microscope (SEM). Elemental analysis was performed using an Oxford energy dispersive X-ray spectrometer attached to the SEM

Results

1. IVD Aluminum

The IVD sample appeared to have lost coating at one corner (Figure 1). The substrate of the IVD sample was found to be very rough on a microscopic scale (Figure 2). EDS analysis showed that the surface was principally iron (Table 1). A significant amount of aluminum was also detected. The aluminum was found to exist as discrete regions or particles held on the iron (steel) substrate by the rough surface. The outer surface of the coating is shown in Figure 3. An oblique view of the coating is shown in Figure 4.

2. Alumiplate

The Alumiplate sample had a smaller amount of exposed substrate than the IVD aluminum sample (Figures 5). The substrate was seen to be rough, but fewer areas of aluminum were seen than on the IVD sample substrate (Figure 6). This was consistent with the EDS compositional analysis, which showed some aluminum on the iron surface, but less than for the IVD samples (Table 2). The outer surface of the coating is shown in Figure 7. The inner surface of the separated coating showed particles of iron (Figure 8). An oblique view of the coating is shown in Figure 9.

Conclusions

The coatings for both systems spalled at the coating/steel bond line. Due to the microscopically rough morphology of these bond lines, separation involved leaving some aluminum on the steel substrate, while a smaller amount of iron was pulled off with the coating. The Alumiplate had a cleaner separation than the IVD aluminum.

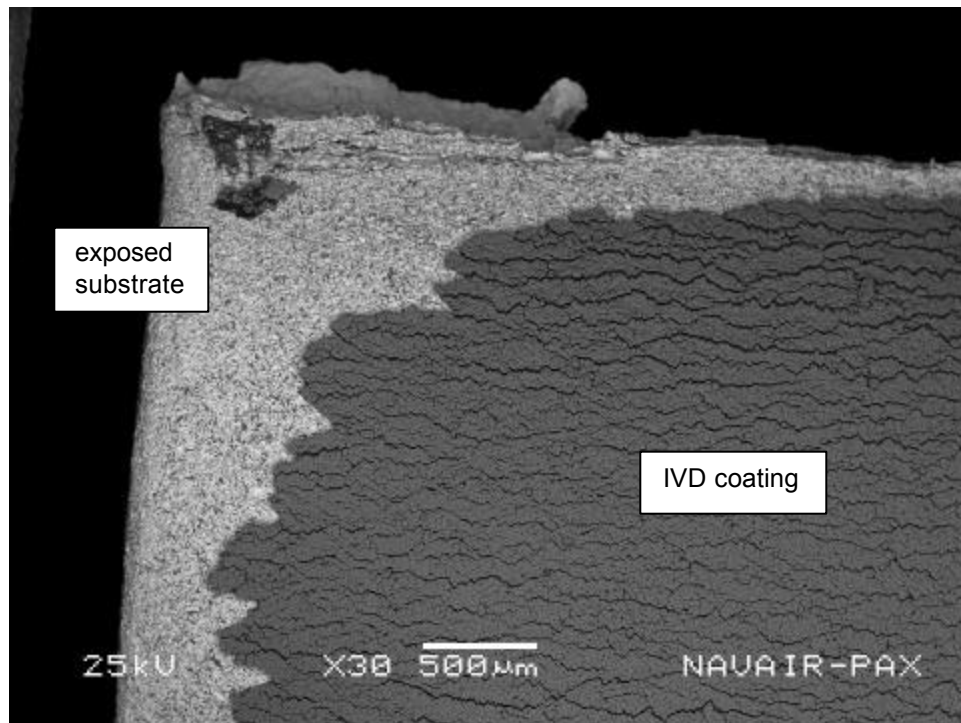


Figure 1. Low magnification SEM image of IVD adhesion test sample.

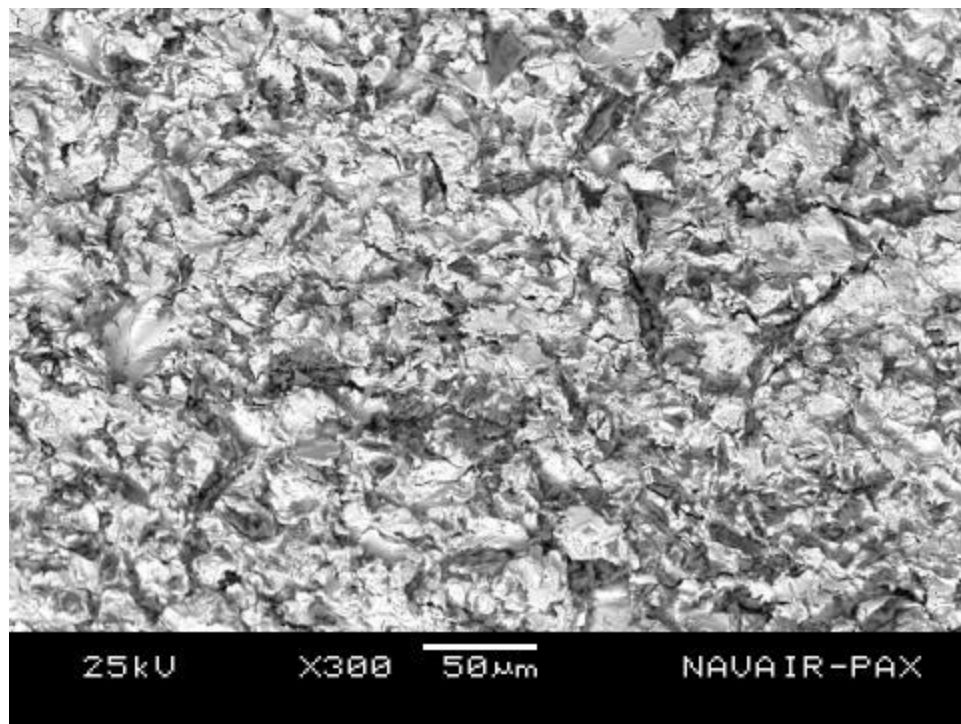


Figure 2. Backscattered SEM image of the IVD sample exposed substrate. Darker appearing regions are aluminum, lighter areas are iron (steel).

	Exposed Substrate (wt%)	IVD Coating (wt%)
Al	22	72
Fe	60	1
Cr	1	3
Mn	<1	...
Si	2	...
O	11	17
C	2	7

Table 1. EDS compositional results for the IVD Aluminum coating adhesion sample.

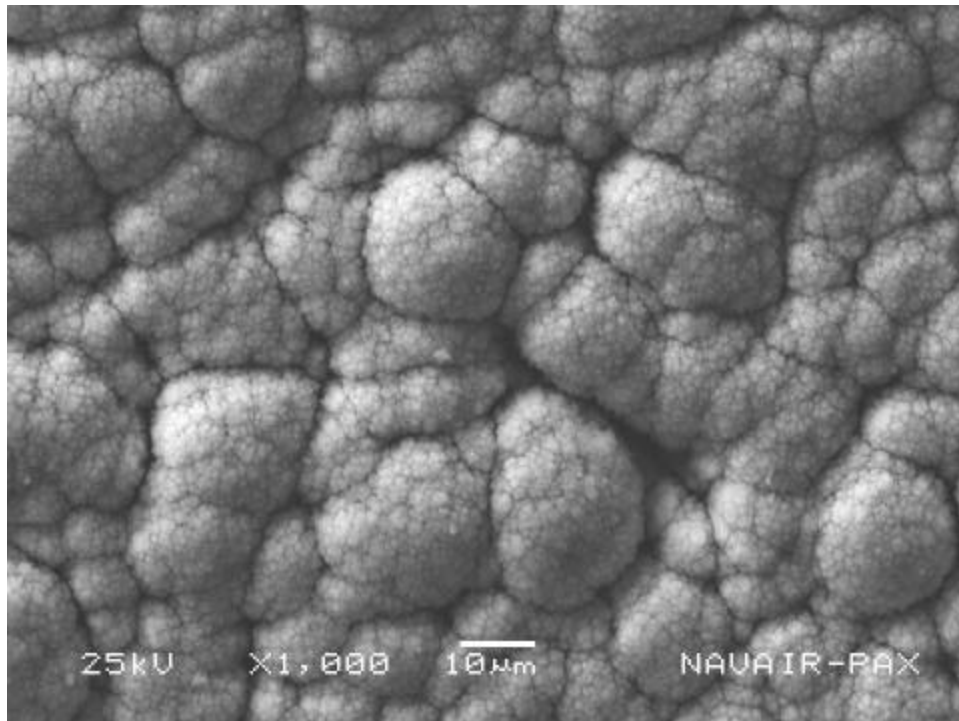


Figure 3. Outer surface of IVD aluminum coating.

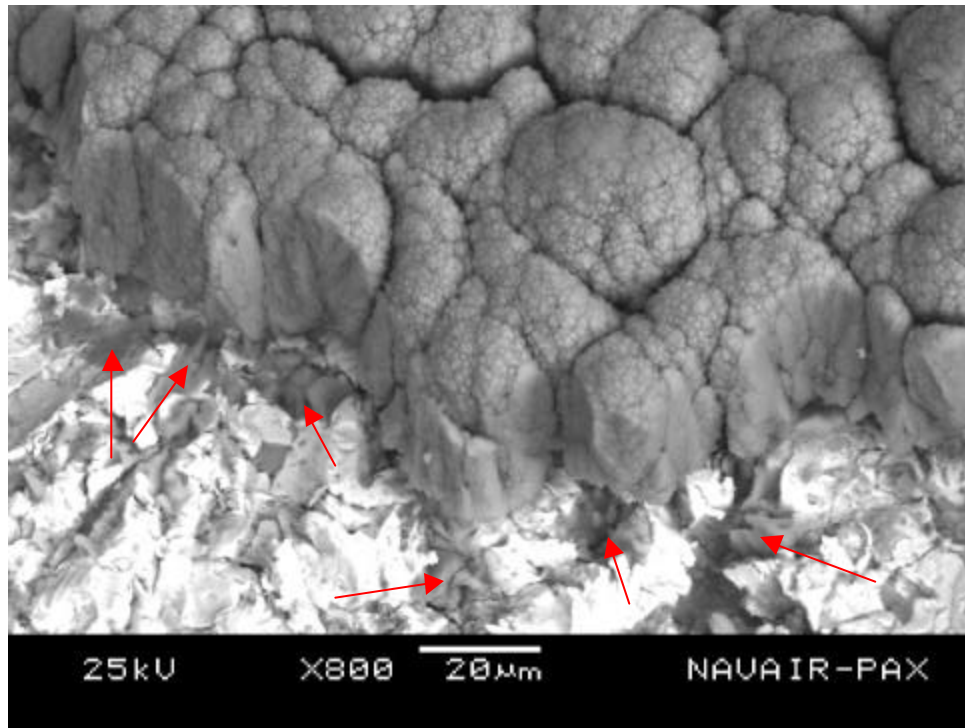


Figure 4. Oblique view of fractured IVD coating and exposed substrate. Remnant aluminum left on the steel substrate can be seen (arrows).

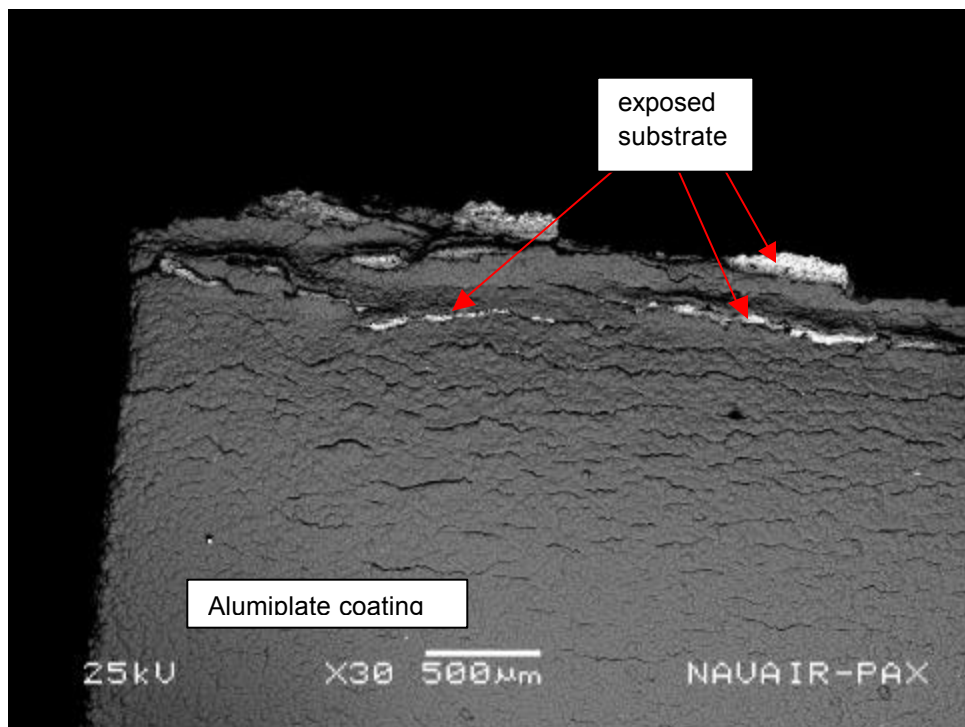


Figure 5. Low magnification SEM image of Alumiplate test sample.

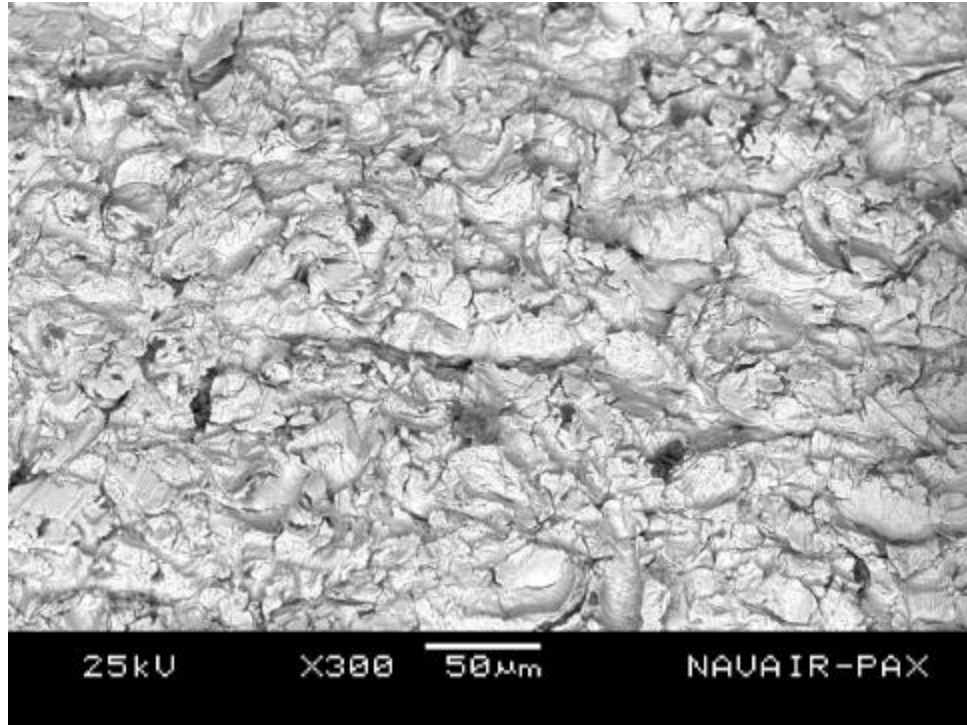


Figure 6. Backscattered SEM image of the Alumiplate sample exposed substrate. Some dark regions indicating aluminum are present, but fewer than on the IVD substrate (compare with Figure 2).

	Exposed Substrate (wt%)	Alumiplate Coating (wt%)
Al	11	78
Fe	86	1
Cr	1	2
Mn	<1	...
O	...	13
C	2	6

Table 2. EDS compositional results for the Alumiplate coating adhesion sample.

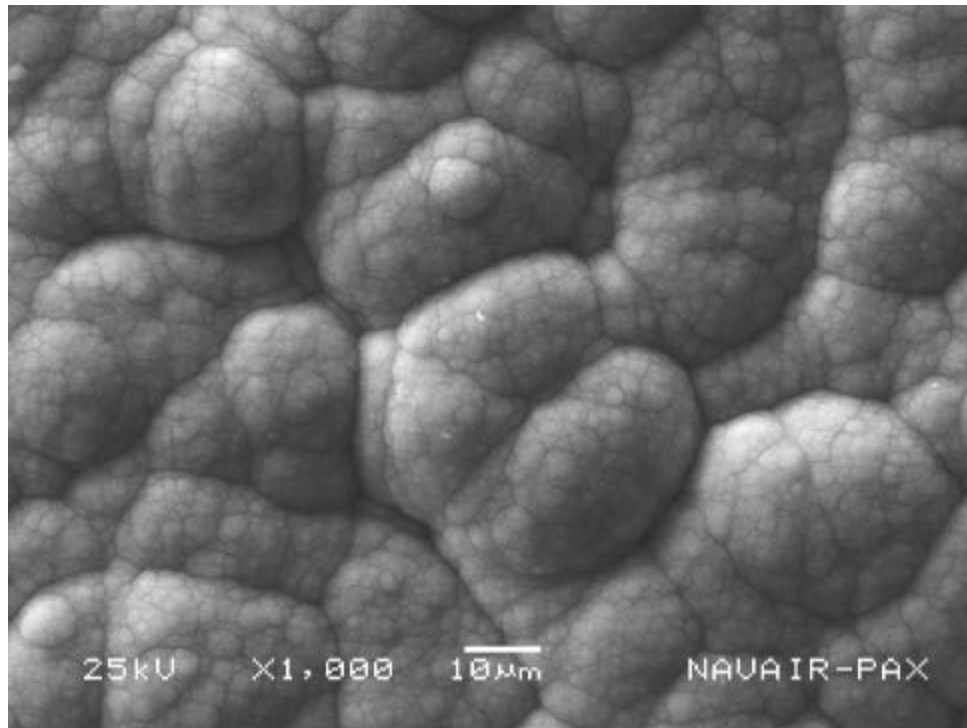


Figure 7. Outer surface of Alumiplate coating.

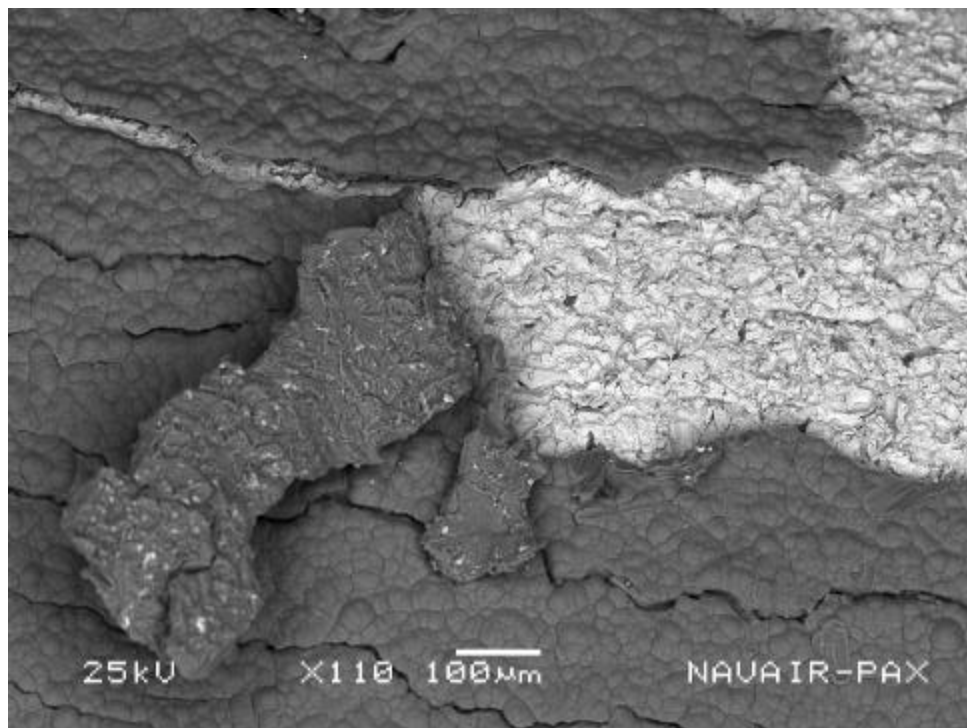


Figure 8. Aluminplate coating peeling away from the substrate. The underside of the peeled coating shows bright flecks that correspond to iron particles pulled from the substrate.

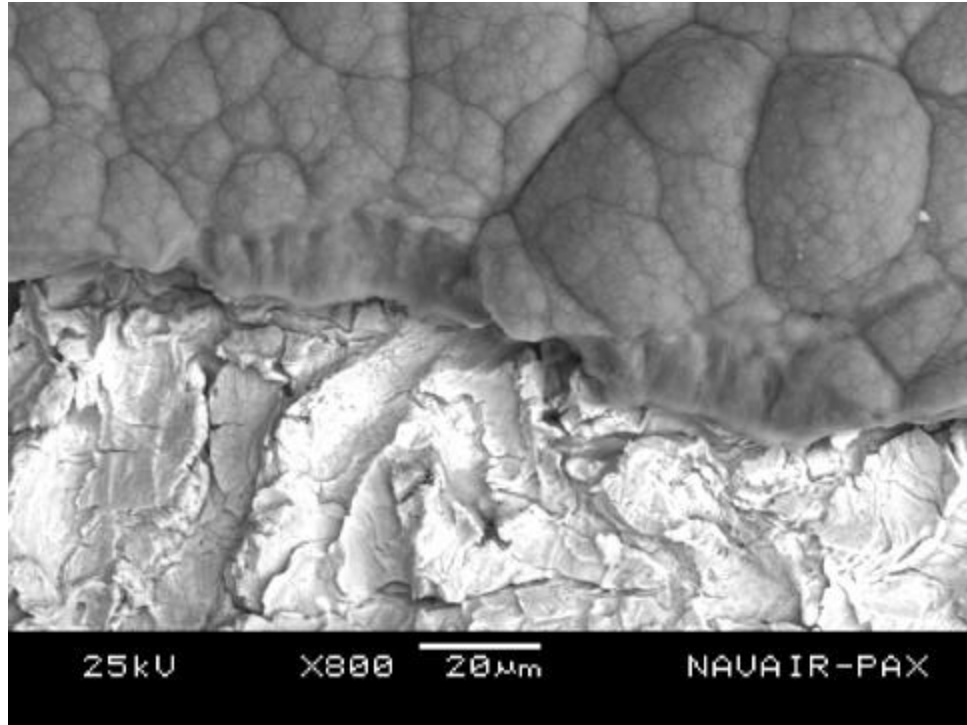


Figure 9. Slightly oblique view of fractured Alumiplate coating and exposed substrate. Little remnant aluminum can be seen on the steel substrate (compare with Figure 4).

APPENDIX D

This Appendix includes the analysis report generated for H-E and H-RE tensile specimens for Phase I of this project. Analysis was conducted by NAWCAD Materials Engineering Division.

Cadmium Alternatives Hydrogen Embrittlement/Re-embrittlement Failure Analysis

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30 September 2005

Introduction

Samples were examined in support of hydrogen embrittlement and re-embrittlement tests in the JTP for Cadmium Alternatives for High Strength Steel Structural Components. These samples consisted of steel notched round bars subjected to rising step load tensile tests in a variety of test fluids. Several bars tested in sustained load in air were also examined. Request was for an examination of the fracture surfaces in order to determine the fracture mode and location of origin.

The sample material was 4340 steel. Fluid-tested samples were coated with:

Alumiplate (3 samples)
Sputtered Aluminum (9)
IVD Aluminum (6)
LHE Cadmium (3)
Boeing Acid Zn-Ni (12)
Dipsol Zn-Ni (5)

Air-tested samples were coated with:

Brush Zn-Ni (2)
Dipsol Sn-Zn (2)

Samples consisted to two mating halves from each test bar. One half of each sample was ultrasonically cleaned in detergent and water. Samples were examined using an stereo optical microscope. The fracture surfaces of the cleaned samples were photographed using a Polaroid Digital Microscope Camera (DMC). These fracture surfaces were then examined at high magnification using a JEOL 6460LV scanning electron microscope (SEM). All samples were optically examined and photographed. Some samples were determined through optical microscopy to be morphologically equivalent to others; in such a case only one of the samples was examined in the SEM.

Results

The majority of specimens displayed a characteristic pattern of fracture morphology. This consisted of (1) a single region of intergranular fracture originating at the surface; (2) a dimpled overload region; (3) a tensile shear lip around the edge of fracture; (4) extensive secondary cracks. This type of failure is shown in Figures 1-3. The intergranular areas varied widely in size.

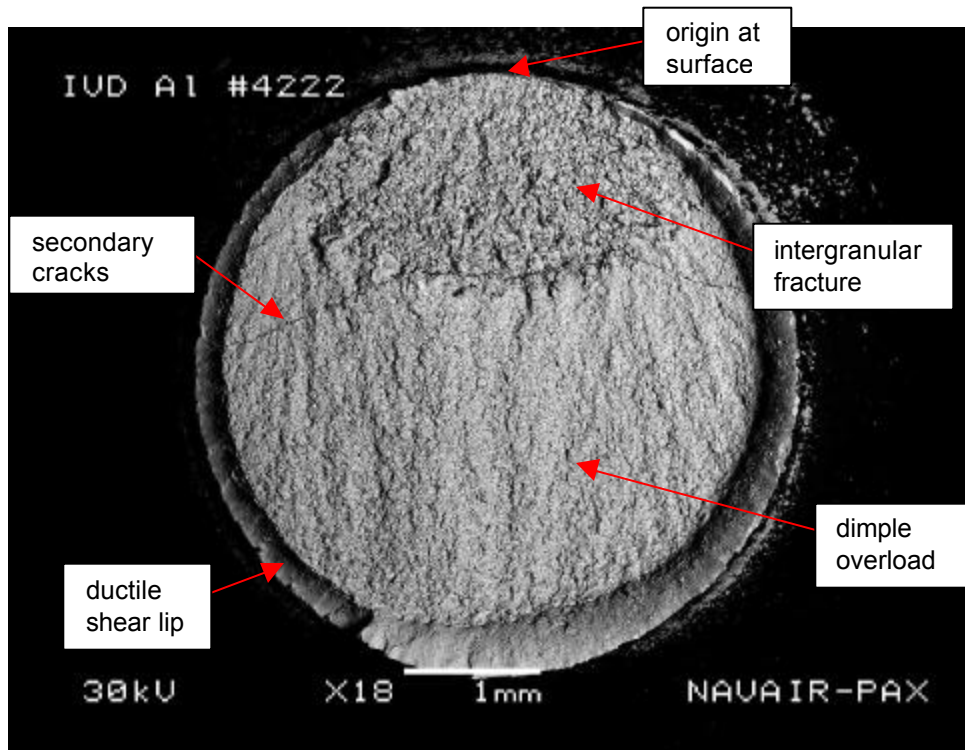


Figure 1. IVD aluminum sample #4222 fracture surface. This sample displays the type of surface most common to the test bars examined. (Reagent water test)

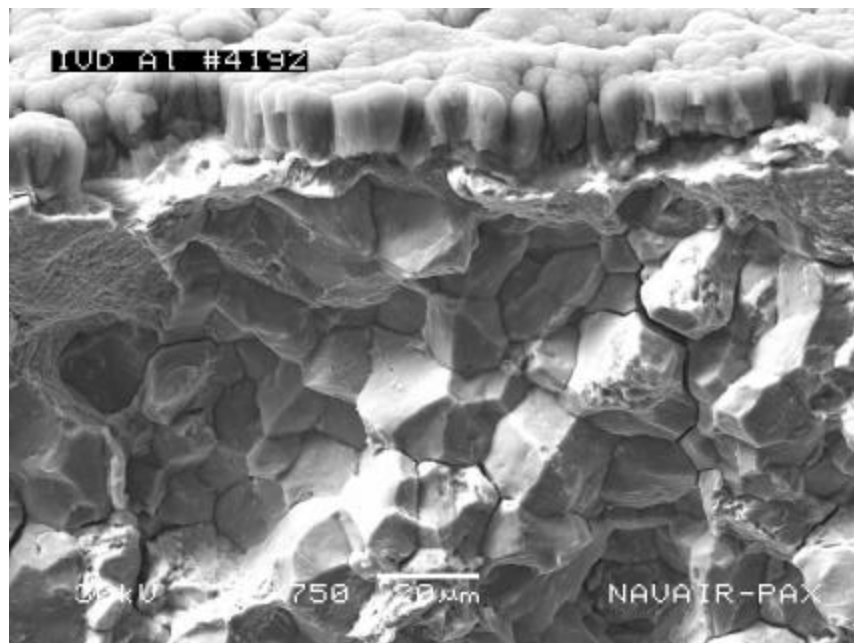


Figure 2. Origin of intergranular fracture region at surface of IVD aluminum sample #4222. The intergranular fracture shows clear grain boundary separation. The coating appears in cross-section along the top of the image.

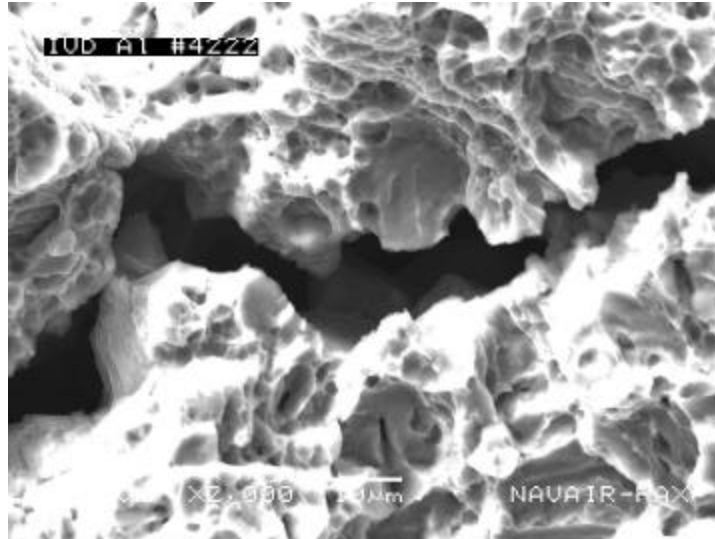


Figure 3. Overload region of IVD aluminum sample #4222, showing ductile dimples covering the surface, and a large secondary crack. The internal surface of the crack appears to be intergranular (brittle), and does not display dimples.

Some of the fractures were essentially the same as that described above, with the exception of having more than one intergranular fracture area, as shown in Figure 4.

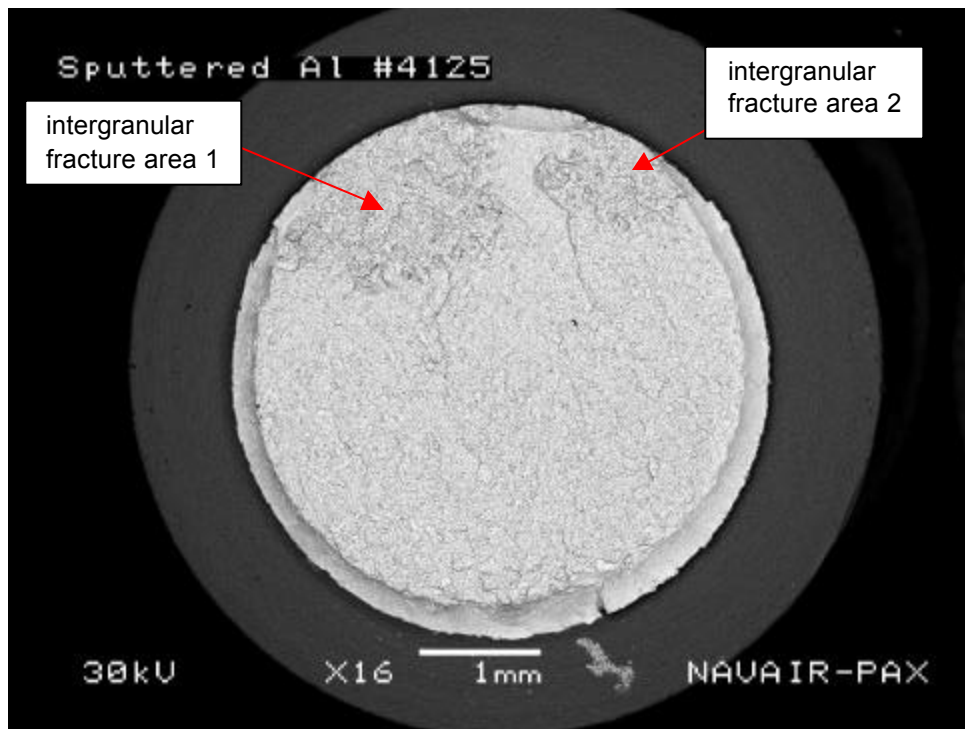


Figure 4. Sputtered aluminum sample #4125, showing two intergranular fracture areas. (Propylene glycol test)

A number of the sputtered aluminum samples displayed a surface that had a large, irregularly shaped intergranular area, as well as an overload area showing a mixed type of morphology – with both ductile dimples and facets suggestive of brittle fracture (Figures 5-7).

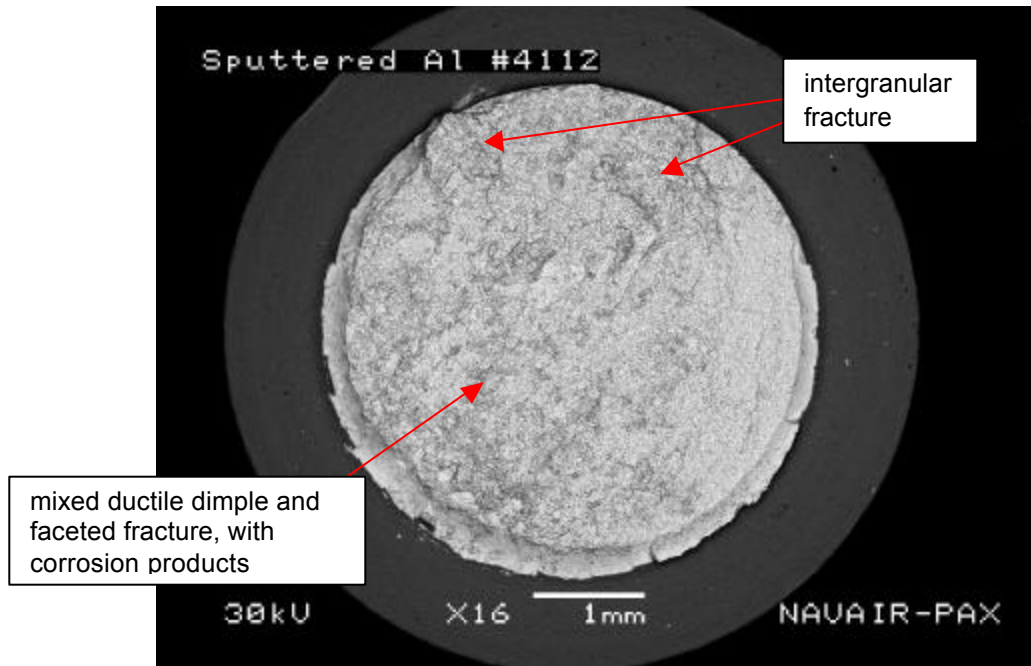


Figure 5. Sputtered aluminum sample #4112, showing an irregular intergranular fracture area at the top, and mixed ductile dimple / faceted surface area.

(Sea water test)

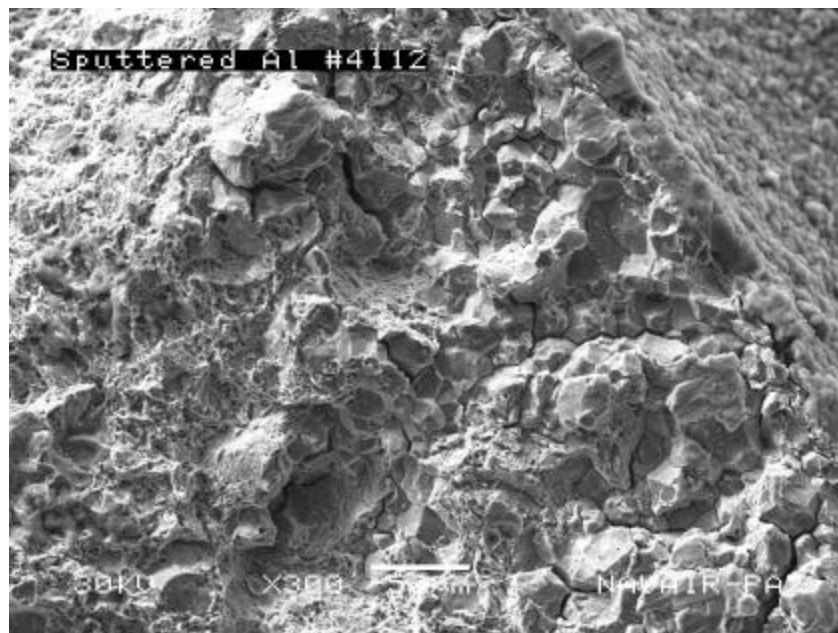


Figure 6. Intergranular fracture at surface of sputtered aluminum sample #4112.

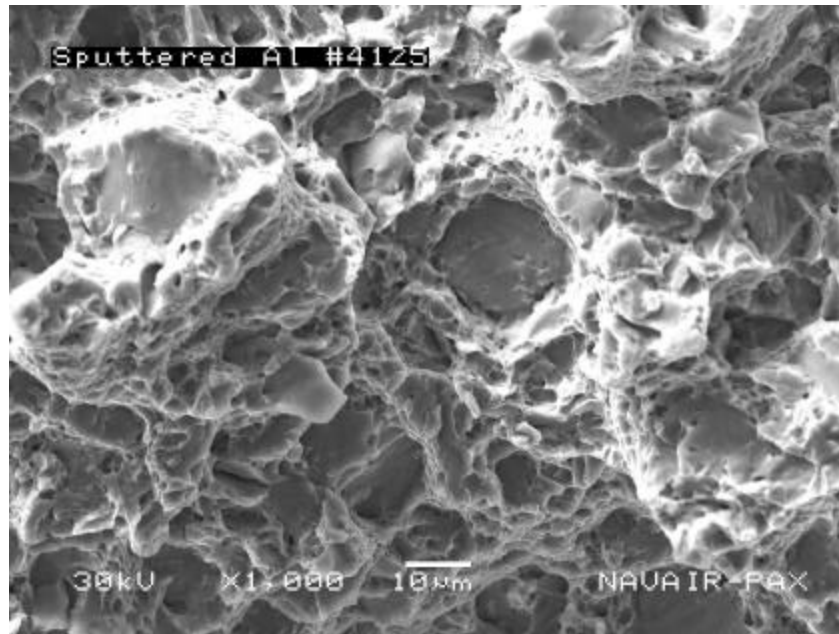


Figure 7. Overload fracture area of sputtered aluminum sample #4112. Dimples characteristic of ductile overload are mixed with some smooth facets suggestive of brittle fracture.

Some samples displayed small amounts of distinct intergranular fracture, but the intergranular areas did not originate at the surface (Figure 8).

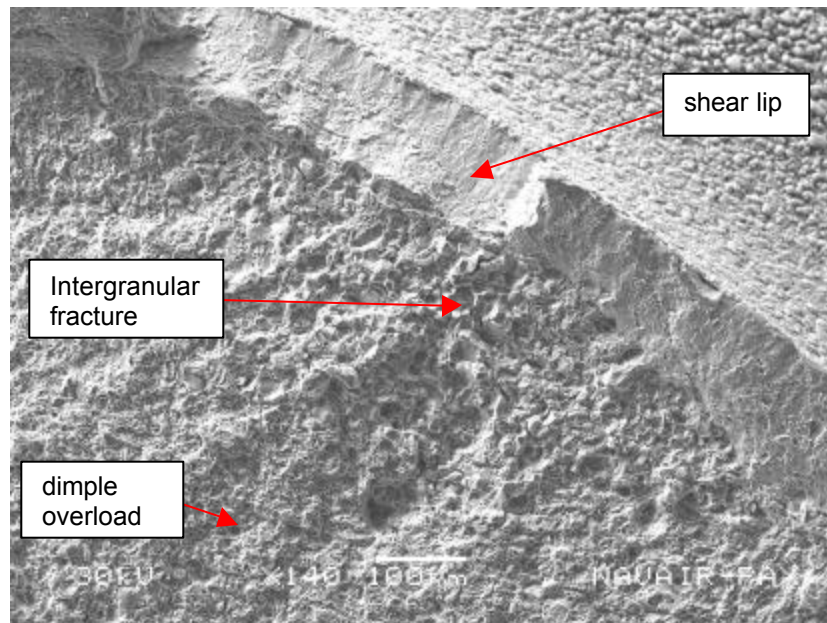


Figure 8. Intergranular fracture area of Brush Zn-Ni sample #4163. The intergranular area does not originate at the surface; there is a substantial shear lip in between. (tested in air)

One sample (Dipsol Zn-Ni sample #1240) had small patches of intergranular fracture all around the edge of the failed surface (Figures 9 and 10).

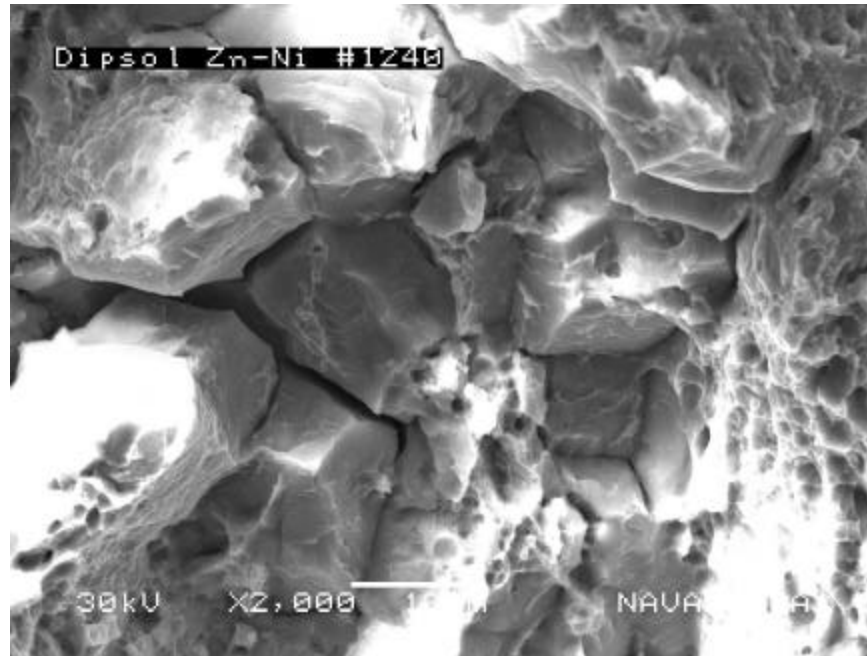


Figure 9. Dipsol Zn-Ni sample #1240, showing a small patch of intergranular fracture. Small areas of similar morphology were found in numerous places around the edge of the sample. (Propylene glycol test)

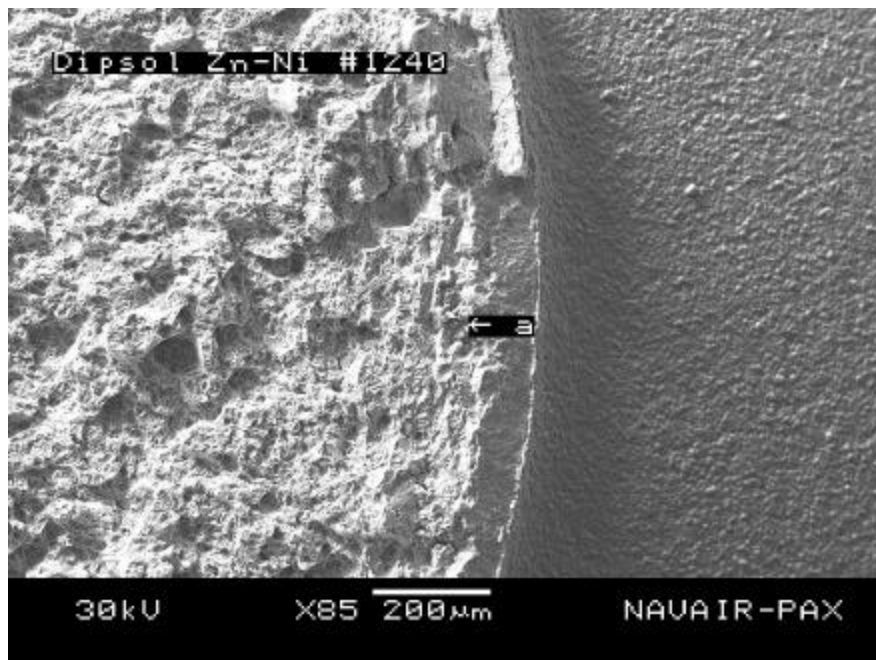


Figure 10. Location of surface shown in Figure 9. The intergranular regions were near, but not at the surface.

Finally, two samples did not display any morphology that could definitively be called intergranular (Brush Zn-Ni 4057 and LHE Cd 4137).

The samples can be classified as follows:

Type A – single large IGF (intergranular fracture region) initiating at the surface

Type B – multiple large IGFs initiating at the surface

Type C – single small IGF initiating at the surface

Type D – irregular IGF with mixed ductile / faceted overload

Type E – IGF region initiating below the surface

Type F– Multiple small IGF regions located around the fracture edge

Type G – No IGF (surface fully ductile overload, no surface initiation)

Summary of Sample Fracture Surface Types

Fluid-tested Samples

Alumiplate (3 samples)

4052	E
4078	C
4097	C

Sputtered Aluminum (9)

4056	A
4063	A
4107	A
4112	D
4123	A
4125	D
4200	A
4227	B
4713	D

IVD Aluminum (6)

1	A
4172	A
4188	A
4192	A
4222	A
4249	A

ESTCP WP-0022 Appendix D

LHE Cadmium (3)

4075	A
4081	A
4137	G...sample displayed some facets potentially indicating brittle fracture

Boeing Acid Zn-Ni (12)

411	A
4055	A
4066	B
4079	A
4088	A
4126	A
4150	A
4167	A
4184	A
4185	B
4193	A
4216	B

Dipsol Zn-Ni (5)

1119	A
1209	A
1240	F
Reagent 1	A
Reagent 4	A

Air-tested Samples

Brush Zn-Ni (2)

4057	G
4163	E

Dipsol Sn-Zn (2)

56	A
185	A

It can be seen from the table that IGF was found on all samples with the exception of only two (LHE Cd #4137 and Brush Zn-Ni #4057). IGF indicates that the samples were embrittled, as does the presence of significant secondary cracking. The transition from IGF to dimple overload is likely the result of changes in the stress state at the front of the growing crack, and does not indicate a change from brittle to non-brittle material. The cracks initiate in an IGF mode, transitioning to dimple rupture as the stress and the crack growth rate increase, even though the material is essentially embrittled throughout. The secondary cracks occur subsequent to sample separation, as demonstrated by the fact that these cracks do not form as mirror image pairs on the mating surfaces. They likely are caused by sonic vibration stresses resulting from the separation of the sample. Their occurrence is supporting evidence that the material has been embrittled.